ANALYTICAL ABSTRACTS

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

3239. **Ultra-micro analysis.** I. P. Alimarin and M. N. Petrikova. *Zavod. Lab.*, 1958, **24** (1), 29–32.—A review covering the years 1952 to 1957, with 47 references. G. S. SMITH

3240. **Air-density corrections for reduction of weighings to vacuum.** E. E. Johnson (Union Carbide Nuclear Co., Oak Ridge, Tenn.). *U.S. Atomic Energy Comm.*, Rep. Y-1181, 1957, 16 pp.—In order to correct for the air displaced by the weights, a formula was devised—$d_a = 10754 \times 10^{-7} + C_t + C_{tw} + C_B$, where d_a is the density of air in g per ml and C_t, C_{tw} and C_B are corrections for the observed dry bulb temp., the observed wet bulb temp. and the barometric pressure, respectively. Specific values of these corrections for the usual temp. and pressure readings are given in tabular form. The value for d_a is then used in the expression $M = m + md_a(1/d_m) - (1/d_w)$ where M is the true mass *in vacuo* in g, m is the apparent mass in g, d_m is the density of the object being weighed in g per ml, and d_w is the density of the weights in g per ml. CHEM. ABSTR.

3241. ***sym.*-Di-*m*- and -*p*-tolylthiovioluric acids as reagents for gravimetric estimation of metals and for salts of organic bases.** P. N. Bhargava, N. Veerabhadriah and B. Satyanarayana (Coll. of Sci., Benares Hindu Univ., Varanasi, India). *J. Indian Chem. Soc.*, 1957, **34** (12), 889–892.—1 : 3-Di-*m*-tolylthiovioluric acid (**I**), m.p. 201°, is prepared by the action of $NaNO_2$ on Na *sym.*-di-*m*-tolylthiobarbiturate; 1 : 3-di-*p*-tolylthiovioluric acid (**II**), m.p. 195°, is prepared similarly. A 2% soln. of **I** or **II** in acetone is added to a standard soln. of the metal salt, and the resulting ppt. is filtered off, washed with acetone, and dried. Only silver, ferrous and uranyl ions give over 99% pptn.; no indication of errors, interference, or standard conditions is given. P. M. SORGO

3242. **Re-conversion of potassium tetraphenylboron.** H. Reimers (Univ. Graz, Austria). *Chemiker Ztg*, 1957, **81**, 357–359.—Residues of $KB(C_6H_5)_4$ obtained in the determination of K are re-converted to $NaB(C_6H_5)_4$ by dissolution in acetone containing a small amount of H_2O and subjected to the action of an acidic cation exchanger in the sodium form. The exchanger is regenerated by aq. NaCl. Yields close to 100% are attainable. CHEM. ABSTR.

3243. **The preparation of high-purity calcium oxide.** R. N. Roberts (Univ. Calif., Los Alamos, N. Mex.). *U.S. Atomic Energy Comm.*, Rep. LA-1936, 1957, 41 pp.—A method is described for the preparation of high-purity CaO required as base matrix of the standards used in the spectrochemical analysis of either Ca or CaO. Double-distilled Ca containing less than 0·2% of metallic impurities was used as the starting material. The method consists in the purification of aq. $CaCl_2$ soln. by modified standard procedures for chemical separations, pptn. of Ca oxalate by the use of carefully purified reagents, and ignition of the oxalate to the oxide under non-contaminating conditions. Special apparatus, developed after numerous tests, to eliminate all possible sources of contamination, such as gold crucibles, platinum ware and titanium and tungsten carbide mortars, are described for the ignition of the Ca oxalate and pulverisation of the resulting oxide in the preparation of the low-level impurity standards. CHEM. ABSTR.

3244. ***p*-Ethoxychrysoidine - oxidising agent systems as new oxyadsorption indicators.** F. Sierra and G. Asensi (Murcia Univ., Spain). *An. Real Soc. Esp. Fis. Quim., B*, 1957, **53** (9–10), 625–634.—The mechanism of action of oxyadsorption indicators (redox indicators in heterogeneous phase) is discussed. Examples are given of the use of *p*-ethoxychrysoidine in association with iodine or vanadate, as an indicator of this type, in the determination of I^- in acid medium, and in the presence of Cl^- or Br^-, by titration with $AgNO_3$ (0·1 *N* or 0·01 *N*). L. A. O'NEILL

3245. **Analysis for industry.** L. Erdey (Dept. Anal. Chem., Tech. Univ., Budapest). *Ind. Chem. Mfr*, 1957, **33**, 523–525.—The applications of lucigenin and luminol as chemiluminescent indicators are reviewed.

3246. **Drying and decomposition of sodium carbonate.** A. E. Newkirk and I. Aliferis (General Electric Res. Lab., Schenectady, N.Y., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 982–984.—An exhaustive thermogravimetric study of Na_2CO_3 in various containers over the range 25° to 1040° is made. Even at 500°, weight losses in siliceous vessels are appreciable and the use of platinum is strongly recommended. D. A. PANTONY

3247. **Basic behaviour of molecules and ions in acetic anhydride.** C. A. Streuli (American Cyanamid Co., Stamford, Conn., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 997–1000.—Titration of neutral and anionic bases in acetic anhydride showed that a linear relationship exists between $pK_a(H_2O)$ values and half-neutralisation potentials. The relationship for neutral bases differs from that for ions; anions are, relative to uncharged molecules, stronger bases in acetic anhydride than they are in water. Halide salts can be directly titrated in the solvent, and chlorides and iodides may be resolved. Anions that form weak acids in H_2O are levelled in basic strength in acetic anhydride soln. G. P. COOK

3248. **Use of propionic acid as a solvent in alkalimetry in non-aqueous medium.** C. Hennart and E. Merlin (Orgasynthèse, Lab. de Vitry, Seine, France). *Chim. Anal.*, 1958, **40** (1), 20.—Titration

curves are given for the potentiometric determination of anhydrous Na acetate and pyridine, with 0·02 *N* $HClO_4$ in anhydrous acetic and propionic acids, a glass electrode, and methyl violet as indicator. It is shown that in acetic acid the titration curve diverges from the equivalence point when the indicator has only reached a green colour, but that in propionic acid this divergence does not begin till the indicator has reached the full yellow. Propionic acid is therefore advocated for all alkalimetric titrations in non-aqueous media in preference to acetic acid. R. E. ESSERY

3249. **The EDTA titration: nature and methods of end-point detection. II.** A. J. Barnard, jun., W. C. Broad and H. Flaschka (J. T. Baker Chem. Co., Phillipsburg, N.J.). *Chemist Analyst*, 1957, **46** (1), 18–28.—Information is given on the visual use of *oo'*-disubstituted azo dyes, *o*-monosubstituted azo dyes, phthalein derivatives, and triphenylmethane and anthraquinone dyes. (109 references.) (*Cf. Anal. Abstr.*, 1957, **4**, 2073.)
N. E.

3250. **Potentiometric and amperometric titrations with tetraphenylboron compounds. I. Argentimetric determinations.** A. Heyrovský (Lab. II. Intern. Hosp., Charles' Univ., Prague). *Chem. Listy*, 1958, **52** (1), 40–42.—*Procedure* (*i*)—Organic bases, K^+, NH_4^+, Rb^+ and Cs^+ can be pptd. with excess of Na tetraphenylboron (**I**), the addition product is isolated, washed and dissolved in acetone and titrated argentimetrically. Interference is caused by Hg^+, Hg^{2+}, Ag^+ and Tl^+, since these metals form similar addition products. Some other interfering ions can be masked. *Procedure* (*ii*)—The sample is pptd. with a soln. of **I**, the addition product is filtered off and the excess of the volumetric reagent in the filtrate is determined argentimetrically. *Procedure* (*iii*)—Titration of the excess of **I** without removing the ppt. yields less accurate results. All titrations were carried out over a wide range of pH (from 0·1 *N* Na acetate to 0·2 *N* HCl), and concn. of 0·1 *N* to 0·005 *N* $AgNO_3$ and 0·03 *N* to 0·005 *N* **I** were used; up to 90% of acetone in the soln. does not interfere. The accuracy is ± 0·1% (potentiometric indication), ± 1 to 2% (amperometric indication, with a dropping-mercury electrode and the use of a short circuit system). All titrations were carried out in a vol. of 20 to 100 ml. With the use of the described procedure, K^+, Tl^+ and 12 organic bases were determined. J. ZÝKA

3251. **Coulometric analysis.** L. E. Smythe (A.E.R.E., Harwell, England). *Ind. Chem. Mfr*, 1957, **33**, 501–505.—The principles of coulometry and recent developments are outlined. (72 references.)

3252. **The distribution of ions in exchange columns.** C. Rocchiccioli (Lab. de Recherches Micro-anal., 11, rue Pierre Curie, Paris). *Mikrochim. Acta*, 1958, (1), 124–136 (in French).—A vertical cation-exchange column (30 cm × 35 mm) was divided into 8 sections by means of cylindrical muslin bags. After conversion of the resin into the acid form, 0·1 *N* soln. of metal chlorides (Na, NH_4^+, Mg^{II}, Zn^{II}, Ni^{II}, Cd^{II}, Fe^{III}, Al^{III}, Th^{IV}) were added to the column in separate experiments. The excess was washed out with water, the bags of resin were withdrawn and each section was separately eluted with HCl. The amount of metal collected in each section was determined. All the metals were found in the first section, Na, NH_4^+, Ni and Cd in the second, and Mg, Zn, Fe and Al in all sections, especially the first and sixth. It is suggested that these findings, which are independent of atomic wt. and valency, are related to the formation of solvates.
G. BURGER

3253. **Electrophoresis, past, present and future.** A. Tiselius (Biochem. Inst., Univ. of Uppsala, Sweden). *Clin. Chim. Acta*, 1958, **3** (1), 1–9 (in English).—A brief review of developments and future possibilities of electrophoresis.
H. F. W. KIRKPATRICK

3254. **Recent developments in polarographic analysis.** G. W. C. Milner and L. J. Slee (Anal. Chem. Group, A.E.R.E., Harwell, England). *Ind. Chem. Mfr*, 1957, **33**, 494–500.—A review. (26 references.)

3255. **Square-wave polarography. IV. An introduction to the theoretical aspects of square-wave polarography.** G. C. Barker, R. L. Faircloth and A. W. Gardner (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 1786, 1958, 38 pp.—Expressions are derived for the alternating part of the current density at the surface of a dropping-mercury electrode when the electrode potential has a square-wave component and the electrode reaction is (*a*) completely reversible, (*b*) slightly irreversible and (*c*) highly irreversible. The variation of wave height with potential is considered for each type of reaction, and formulae are given which can be used to calculate the max. wave height and the half-wave width. The use of an analytical type of square-wave polarograph for the determination of diffusion coefficients and rate constants and for the identification of reversible and irreversible reactions is also discussed. K. A. PROCTOR

2.—INORGANIC ANALYSIS

3256. **5-Chloro-2:4-hydroxyacetophenone as a reagent in inorganic analysis. Colour reactions with cations.** Samir K. Banerji and Arun K. Dey (Chem. Lab., Univ. of Allahabad, India). *Z. anal. Chem.*, 1958, **159** (4), 293–294 (in English).—A 1% soln. of 5-chloro-2:4-hydroxyacetophenone in ethanol has been found to be a very sensitive colour reagent for ferric and uranyl ions. In neutral medium, $FeCl_3$ gives a black colour and uranyl nitrate a brown-yellow colour, but no change in colour is observed in acid or alkaline solution. The reagent is indifferent to most other metals, and the possibility of using it in micro-quant. colorimetric analysis for the two ions is being investigated.
S. M. MARSH

3257. **Back-titration with mercuric nitrate in alkaline medium: analysis of binary mixtures of barium, strontium or magnesium together with lead, cobalt, nickel or copper.** H. Khalifa (Vienna Univ., Austria). *Z. anal. Chem.*, 1958, **159** (6), 410–414 (in English).—Two identical mixtures, one at a lower, and the other at a higher, pH, are back-titrated potentiometrically after excess of EDTA (disodium salt) has been added to them.
E. G. CUMMINS

3258. **Chelometric titrations of metal ions with potentiometric end-point detection. Ethylenediaminetetra-acetic acid.** C. N. Reilley, R. W. Schmid and D. W. Lamson (Dept. of Chem., Univ. of N. Carolina, Chapel Hill, U.S.A.). *Anal. Chem.*, 1958, **30** (5), 953–957.—Cations are titrated potentiometrically with EDTA by the use of the mercury electrode discussed by Reilley and Schmid (*cf. Anal. Chem.*, 1958, **30**, 947). The ions may be (*a*) titrated directly with EDTA in an ammonia, acetate or triethanolamine buffer or in acid soln. or (*b*) complexed with an excess of EDTA, which is back-titrated with standard Zn^{2+} or Cu^{2+} in an acetate buffer or with Zn^{2+} in an ammonia buffer. The electrode and typical titration graphs are illustrated. By suitable intermediate adjustment of pH, a mixture of Bi^{3+} (pH 1·2 to 2), Cd^{2+} (4) and Ca^{2+} (8) can be determined by this method. Results for the titration of 29 cations, which can be determined with a precision of up to ± 0·4%, are presented. D. A. PANTONY

3259. **Spectrophotometric studies of chelates of 8-quinolinol [8-hydroxyquinoline] in some water-miscible organic solvents. Photometric titrations with 8-quinolinol.** W. G. Boyle, jun., and R. J. Robinson (Univ. of Washington, Seattle, U.S.A.). *Anal. Chem.*, 1958, **30** (5), 958–961.—The titrations of Cu^{2+}, Zn^{2+}, Cd^{2+}, Ni^{2+}, Pb^{2+} and UO_2^{2+} have been investigated photometrically in dioxan - *n*-propanol and in dimethylformamide, with 8-hydroxyquinoline as titrant. Titrations of Cu^{2+} and Zn^{2+} could be made with a precision of 1 or 2 parts per 1000, but titration of the other cations was unsatisfactory. A high-absorbance reference procedure was used. K. A. PROCTOR

3260. **Chromatographic study of metal ammines.** E. J. Singh and Arun K. Dey (Chem. Lab., Univ. of Allahabad, India). *Z. anal. Chem.*, 1958, **159** (4), 290–292 (in English).—The chromatographic behaviour of the ammine complexes of Cu^{II}, Ag, Cd^{II}, Ni^{II} and Co^{II} have been studied. Solutions of the salts of these metals containing various amounts of NH_4Cl and aq. NH_3 were spotted on filter-paper strips and 50% ethanol was used as the developing solvent. Progressive increase in the concn. of aq. NH_3 increased the R_F values for Cu and Ni, and decreased these values for the complexes of Ag, Cd and Co, but the R_F values tended finally to become constant. An excess of NH_4Cl gave improved chromatograms. S. M. MARSH

3261. **Electro-migration on paper in the separation of ions. VIII.** Hari Gopal Mukerjee (Scottish Church College, Calcutta-6, India). *Z. anal. Chem.*, 1958, **159** (4), 287–290 (in English).—The electromigration of Os^{IV}, its separation from Hg, Cu, Cd and Bi, the electro-migration of UO_2^{2+} and its separation from Pt, Pd, Bi and Fe, and the separation of As and Sb from Pt, Pd, Ru and Au with different electrolytes of various concn. are described. Gold and platinum-group elements cannot be separated electrophoretically from Sn as there is immediate formation of a ppt. or separation of metal when gold or platinum-group salts are mixed with tin salts. S. M. MARSH

3262. **Chemical analysis of copper in Zircaloy, silicon in uranium - silicon alloys and niobium in uranium - niobium alloys.** E. B. Read, P. R. Hicks, H. M. Lawler, E. Pollock, H. M. Read and L. Zopatti (Nuclear Metals Inc., Cambridge, Mass.). *U.S. Atomic Energy Comm.*, Rep. NMI-1178, 1957, 17 pp.—The neocuproin method gave low results for Cu in Zircaloy, owing to interference by Sn in the alloy. Increasing the amount of hydroxylamine hydrochloride employed in the analysis resulted in improved recovery of Cu. Complete recovery was obtained by adding 10 ml of 20% hydroxylamine hydrochloride, 10 ml of 30% Na citrate and 10 ml of 0·1% neocuproin soln. to an acid soln. of the sample. The soln. is mixed, allowed to stand for 5 to 10 min., adjusted to a pH of 5 and extracted with $CHCl_3$. The extinction of the extract is measured at 457 mμ. In the application of the molybdosilicic acid method to the determination of Si in uranium alloys, low results are obtained when the "as cast" alloy is dissolved in HNO_3 followed by HF, owing presumably to evolution of a gaseous silicon compound. Preliminary oxidation of the sample in a muffle-furnace at 800° enabled it to be dissolved without loss of Si. A gravimetric tannin method gave very accurate results in the determination of Nb in Nb - U alloys, but requires 2 days for completion of an analysis. As a more rapid procedure, the spectrophotometric method of Pickup (*cf. Anal. Abstr.*, 1955, **2**, 2707) was investigated and adapted to the analysis. Acceptable agreement with the gravimetric method is obtained. CHEM. ABSTR.

3263. **Some fission-product partition data for butex (dibutyl Carbitol).** H. A. C. McKay, K. Alcock and D. Scargill (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 2221, 1958, 6 pp.—The partition coeff. (*K*) of trace amounts of ^{91}Y and ^{144}Ce between H_2O and dibutyl Carbitol (dibutyl digol) (**I**) determined in the presence of HNO_3 (3 to 9·2 *M*) are listed. The values of *K* are very low, but Ce^{III} is rather more extractable into **I** than is Y. Values of *K* for ^{95}Zr (free from ^{95}Nb) between the same two media, determined in HNO_3, are also given. The curve of $K_{Zr} \times 10^3$ rises steeply from 0·26 at 0·8 *M* HNO_3 to ≃ 80 at 7 *M* and then flattens to 173 at 11·5 *M* HNO_3. The curve is much lower than the corresponding curve for 20% tributyl phosphate - light petroleum. W. J. BAKER

3264. **Chromatographic determination of argon in gas analyses.** O. Harva and A. Keltakallio (Neste Oy Refinery, Naantali, Finland). *Suomen Kem., B*, 1957, **30** (12), 223–224 (in English).—When gas chromatography on a column of silica gel is applied to a mixture of gases, argon has the same retention time as oxygen; by absorption of the oxygen with pyrogallol or $CrCl_2$ the argon can be determined. The temp. used was 30° and the carrier gas nitrogen. N. E.

3265. **Rapid determination of alkali hydrogen carbonates in the presence of alkali carbonates by a precipitation method.** L. Szekeres and E. Bakács (née Polgár) (Univ. Budapest, Hungary). *Z. anal. Chem.*, 1958, **159** (6), 414–418.—The carbonate mixtures are titrated against NaOH soln. (standardised against $ZnCl_2$ soln.), in the presence of $BaCl_2$ and alkali chloride, with phenolphthalein as indicator. E. G. CUMMINS

3266. **Determination of lithium hydride and lithium hydroxide in commercial-grade lithium hydride.** J. W. Frazer, C. W. Schoenfelder and R. L. Tromp (Univ. California, Livermore). *U.S. Atomic Energy Comm.*, Rep. U.C.R.L.-4944, 1957, 9 pp.—The reaction $LiOH + LiH \rightarrow Li_2O + H_2$ goes to 94 to 95% completion in 30 min. at 400°. When the sample is treated with boiling mercury this

reaction occurs simultaneously with the reaction $2LiH + 2Hg \rightarrow 2LiHg + H_2$. Usually the former reaction goes to about 85% completion before the LiH is used up by the latter reaction. The LiH assay can then be obtained from the difference of evolved H for the reactions with and without mercury. CHEM. ABSTR.

3267. **Quantitative determination of potassium in sodium chloride with sodium tetraphenylboron.** B. Riva (Inst. Appl. Chem., Univ. Cagliari). *Ann. Chim., Roma*, 1958, **48** (1), 50–55.—The K is pptd. as K tetraphenylboron at 0° and pH 2. For the reagent, dissolve 1 g of Na tetraphenylboron in 100 ml of water containing 1 ml of *N* NaOH, add 0·5 g of $Al(OH)_3$ and filter. Dissolve 10 g of the NaCl sample in 200 ml of water, filter, add 30 ml of 0·1 *N* HCl and 50 ml of water, cool to 0°, and add dropwise 20 ml of the reagent cooled to 0°. Filter, wash the ppt. with water, then with a soln. containing K tetraphenylboron, dry at 105° for 30 min., and weigh as K tetraphenylboron. Tables are given of the accuracy of the method under various conditions. Interference is caused by NH_4^+, Rb and Cs. A. G. COOPER

3268. **New method of analysis of copper alloys. [I].** M. Kuhn (Div. des Études Phys.-Chim., Centre Techniques des Industries de la Fonderie, France). *Chim. Anal.*, 1958, **40** (1), 11–19.—After a critical discussion of previous methods of analysis of brass and bronze, in which the Sn is insolubilised with HNO_3, the complete solution of the alloy in a HNO_3 - HBF_4 mixture is advocated, as given for Mn in the 1946 Ed. of the methods of the A.S.T.M. The solution is carried out in Pyrex-glass vessels, which, to minimise attack, are treated with silicone. Silicon and Al with a negligible trace of Fe are dissolved from the glass, and must be estimated on a separate sample. In the solution so obtained, Cu and Pb are removed by electrolysis, and the residual solution, after fuming with H_2SO_4, is made up to a known volume, and Sn, Zn, Fe, Mn, Ni and Sb are determined in aliquots. Suitable methods are briefly described. Other elements determined separately are Al (colorimetrically after removal of interfering elements by electrolysis with a mercury cathode), S (by combustion to SO_2 in a stream of O) and As (nephelometrically after reduction with hypophosphite). R. ESSERY

3269. **Rapid method of determining copper, lead and zinc in polymetallic ores and concentrates.** S. Yu. Faĭnberg, A. A. Blyakhman and L. N. Filatova (State Sci. Res. Inst. of Non-Ferrous Metals). *Zavod. Lab.*, 1958, **24** (1), 18–20.—Lead can be titrated with EDTA in the presence of complex cyanides of Fe, Zn, Cu, Cd, Ni and Co. The subsequent addition of formaldehyde, which reacts rapidly only with the complex of Zn, allows Zn to be determined by EDTA. The sample (0·25 g) is decomposed by treatment with HNO_3 and Br and evaporation, or by treatment with HCl and HNO_3 and evaporation with H_2SO_4, and the solution of the residue in 10 ml of water is treated with aq. NH_3 until $Fe(OH)_3$ starts to precipitate, then with 0·8 g of NH_4HF_2 and 10 ml of 10% KI soln. The Cu is determined by titration of the liberated iodine with $Na_2S_2O_3$ soln. Next, 0·5 g of ascorbic acid is added to reduce Fe^{3+}, followed by 1 g of citric acid and aq. NH_3 added dropwise to give incipient pptn.; and the soln. is treated with 3 ml of conc. aq. NH_3, 7·5 ml of 20% KCN soln., 100 ml of water and 20 ml of a buffer soln. (13·4 g of NH_4Cl and 88 ml of conc. aq. NH_3 diluted to 250 ml). The soln. is heated gently until it is light yellow, then treated with 3 g of NH_4F and boiled for 1 to 2 min. After having been kept at room temp. for 15 to 20 min., the soln. is titrated with EDTA in the presence of Eriochrome black T as indicator until the colour changes from violet to green. A variation of the method allows for the use of hydrazine instead of ascorbic acid. To determine the Zn, the titrated soln. is mixed with formaldehyde soln. in small portions until a violet colour changing to red on stirring develops. The soln. is then titrated with EDTA until a green colour develops. Further additions of formaldehyde followed by titration are made until no further change of colour occurs.

G. S. SMITH

3270. **Rapid analytical methods for metals and minerals. V. Polarographic determination of copper and bismuth in minerals and in iron.** J. Doležal and J. Novák (Inst. Anal. Chem., Charles' Univ., Prague). *Chem. Listy*, 1958, **52** (1), 36–39.—*Procedure*—Dissolve the finely powdered sample (1·5 g, containing about 0·5% of Cu) in a mixture of conc. HCl (5 ml) and HNO_3 (1 ml). Evaporate twice with conc. HCl (2 ml) and heat for 10 min. with H_2O (20 ml) and conc. HCl (2 ml) on a water bath. Cool and dilute the soln. with H_2O to 100 ml. To a 10-ml aliquot in a 25-ml flask add 10 *N* HCl (5 ml), 5 *M* NaH_2PO_2 (3 ml) (for the reduction of Fe^{3+}) and 0·1 *N* $Hg(NO_3)_2$ (0·1 ml), heat at boiling-point till the soln. is decolorised. Cool the soln., add a 1% soln. of gelatin (2·5 ml), dilute to volume with H_2O, remove oxygen with a stream of N (5 min.) and register the polarographic wave from 0 to − 0·6 V. Make a parallel determination with a standard containing 0·5 mg of Cu. As little as 0·01% of Cu in iron can be determined with high accuracy. Interference is caused by Mo and Ti, and an excess of Bi or Sb. J. ZÝKA

3271. **Photometric determination of silver in the presence of copper.** G. Ciuhanda and V. Giuran (Inst. Hygiene, Lab. Toxikol., Timişoara, Romania). *Z. anal. Chem.*, 1958, **159** (4), 250–256.—The procedure described is based on the formation of a stable silver sol by the alkaline reduction of the *p*-sulphamoylbenzoic acid complex, with CO as the reducing agent. The silver sols are unstable in the presence of an excess of either alkali or CO, when displacement of Ag^+ adsorbed on the sol particles occurs. Experiments showed that the sols could be stabilised by dilution after reduction, and that this stabilisation is due to a reduction of the OH^- concn. The reproducibility of the extinction coeff. of the diluted sols was not satisfactory, and further stabilisation by the addition of various quantities of Cu^{2+} was attempted. The light absorption and stability of the resulting sols was found to vary with Cu concn., and the reduction should therefore be carried out at a constant Cu^{2+} concn. determined by the solubility of basic copper carbonate. *Procedure*—To 40 ml of the test soln., which has been previously neutralised if acidic, is added 0·5 ml of 0·1 *M* $CuSO_4$ followed by 2 ml of 0·1 *M* Na sulphamoylbenzoate, and finally 2 ml of *M* Na_2CO_3 - *M* NaOH (4:1, v/v). After dilution to 50 ml, the mixture is filtered and 20 ml of the filtrate is made alkaline with 2 ml of *M* NaOH. Carbon monoxide (25 ml) (generated from formic acid by the action of H_2SO_4 since a soln. in ethanol or acetone produces unstable sols with non-reproducible extinction coeff.) is then passed through the

soln., which is then shaken intermittently for 30 min. The extinction coeff. of the sol is then measured and the silver concn. is estimated from a calibration curve prepared under similar conditions. If sufficient quantities of Cu are already present in the soln., Na sulphamoylbenzoate is added first and then 20% NaOH soln. until the soln. is alkaline to phenolphthalein; the mixture is finally treated with the Na_2CO_3 - NaOH mixture. The silver content is determined by reference to a calibration curve prepared at a given concn. of Cu. The procedure may be applied to the determination of Ag either alone or in the presence of Cu, over a wide range of silver concn., and is suitable for routine analysis. When considerable quantities of basic copper carbonate are pptd., some Ag is also carried down. In order to obtain reproducibility of the extinction coeff. at the same silver concn., reduction must be carried out at the same temperature.

S. M. MARSH

3272. **Investigation of atomic emissions by flame photometry. I. Flame-photometric properties of silver salts.** E. Pungor and I. Konkoly Thege (Inst. Inorg. Anal. Chem., L. Eötvös Univ., Budapest). *Acta Chim. Acad. Sci. Hung.*, 1958, **13** (3–4), 235–241 (in German).—The flame-photometric determination of Ag has been investigated at 2600° and 1850° by means of a Beckman DU photometer, the relation between atomic emission of Ag and concn. of $AgNO_3$ in the soln. being observed graphically. The effects of ethanol concn. and of different anions were also investigated. The results agreed with those of earlier experiments on the alkali metals (*Anal. Abstr.*, 1957, **4**, 28).

J. S. HETMAN

3273. **Qualitative analysis. III. Separation of the alkaline-earth group.** L. S. Bark (Derby and Dist. Coll. of Technol., England). *Mikrochim. Acta*, 1958, (1), 117–119 (in English).—Loss of the alkaline earths during the group separation can be avoided by pptg. them with 4 *N* H_2SO_4 and ethanol immediately after the silver group. The sulphates are separated by centrifuging, boiled with ammonium acetate to remove any Pb and fused with Na_2CO_3. The melt is boiled with H_2O and the residue dissolved in 4 *N* acetic acid. The cations in the soln. can then be identified by the usual methods.

G. BURGER

3274. **New colour reaction for the detection and determination of beryllium with Chrome blue K.** L. P. Kalinchenko, N. P. Strakhov and I. I. Kalinichenko (Sverdlovsk Med. Inst.). *Zavod. Lab.*, 1958, **24** (1), 22–23.—In ammoniacal soln. (pH 9 to 10) Be gives a sol. blue compound with Acid Chrome blue K (0·25% aq. soln.). Other ions, Cu, Ni, Al, Co, Ba, Mg, Ca, Cd, Zn, etc., that give colours do not interfere if they are combined with EDTA. The reaction is sensitive to 5×10^{-7} g of Be per ml on filter-paper, or to 5×10^{-8} g of Be in a drop of soln. For the former test, a drop of the soln. is placed on filter-paper followed by a drop of an ammoniacal buffer soln. (100 ml of 20% NH_4Cl soln. and 100 ml of 20% aq. NH_3 diluted to 1 litre with water), a drop of 0·1 *N* EDTA (disodium salt) and a drop of the reagent soln. The paper is dried and the colour of the centre of the spot is observed. The max. absorption of the reagent is at 580 to 590 mμ and that of the complex at 600 to 610 mμ. The colour appears at once and attains its max. in 20 to 30 sec. For the determination of Be, a series of standards is prepared from a soln. containing 10^{-6} g of Be per ml. Portions (1·4 to 2·3 ml) are placed in ten tubes and to each are added 1 ml of $CuSO_4$ soln. containing 10^{-4} g of Cu per ml, 1 ml of 0·1 *N* EDTA, one drop of the reagent, 2 ml of the buffer soln. and sufficient water to give a volume of 10 ml. To determine Be in bronze, the sample (0·1 g) is dissolved in 4 ml of dil. HNO_3 (1:2) and the soln. is evaporated to a volume of 1 to 1·5 ml. The soln. is diluted to 100 ml and an aliquot is treated with EDTA, reagent and buffer soln., as described for the standards.

G. S. SMITH

3275. **Acid Chrome dark blue and Acid Chrome blue K as indicators in the complexometric determination of calcium.** T. B. Styunkel' and E. M. Yakimets (S. M. Kirov Ural Polytech. Inst.) *Zavod. Lab.*, 1958, **24** (1), 23–25.—The use of Acid Chrome dark blue and Acid Chrome blue K instead of murexide enables Ca and Mg to be determined on the same sample with the same indicator but at different pH. The sample (carbonate mineral) (0·1 g) is dissolved in the minimum amount of dil. HCl (1:1) and the soln. is neutralised to Congo red with NaOH soln. It is then treated with 5 ml of 2 *N* NaOH and, after dilution to 100 ml and being set aside for 1 to 2 min., the soln. is titrated with 0·1 or 0·01 *N* EDTA (disodium salt) in the presence of a few drops of the indicator until the colour changes from red to bluish-lilac to give the content of Ca. If large amounts of Mg are present, 2 ml of a 2% soln. of sucrose is added before the addition of NaOH. To determine the Mg, the ppt. is dissolved by addition of 5 ml of 2 *N* HCl to the titrated soln., and the titration is continued after addition of 5 to 6 ml of an ammoniacal buffer soln. (pH 10).

G. S. SMITH

3276. **Photometric micro-determination of calcium with picrolonic acid and methylene blue.** D. C. Nonowa (Univ. Sofia, Bulgaria). *Mikrochim. Acta*, 1958, (1), 111–116.—A procedure is described for the determination of from 10 to 80 μg of Ca in the absence of other ions with a mean error of ± 1%. Calcium is pptd. at 50° by the dropwise addition of a 3- to 4-fold excess of lithium picrolonate, with constant shaking. After filtration and washing with ice-cold water followed by anhydrous ether, the ppt. is dissolved in hot water. Chloroform and a slight excess of methylene blue are added, and the whole is shaken vigorously. The chloroform layer is separated and its extinction measured at 643 mμ. The extinctions conform to Beer's law.

G. BURGER

3277. **Experiences with flame spectrometry in determination of calcium and magnesium in a steel-plant laboratory.** K. Abresch and W. Dobner (August Thyssen-Hütte A.-G., Duisberg, Germany). *Arch. Eisenhüttenw.*, 1958, **29** (1), 25–34.—Interferences introduced in the determination of Ca and Mg by the presence of other dissolved substances can be removed or considerably reduced by the addition of a conc. soln. of strontium chloride. In blast-furnace slag the error due to silica is thus made much smaller. Methods are also discussed for the rapid determination of Ca and Mg together, with evaluation of results, possible errors, reproducibility and time requirements. (24 references.)

H. SAWISTOWSKI

3278. **The spectrographic determination of calcium and zirconium in bismuth - uranium alloys.** R. C. Smart and M. S. W. Webb (U.K.A.E.A., Chem. Div., Woolwich Outstation, England). A.E.R.E. Report C/R 2117, 1958, 8 pp.—From

50 to 500 p.p.m. of Ca or Zr in Bi - U alloys can be determined by converting the metals into oxides (ignition of nitrates at 600°), mixing in 10% of La_2O_3 as internal standard, and cementing 5-mg charges to copper electrodes with thermosetting resin. After excitation by a triggered uni-directional discharge, the spectra are recorded by either a Hilger 3-metre grating spectrograph (slit width 0·015 mm, length 20 mm, λ 3200 to 4700 A) or a large automatic quartz instrument (slit width 0·015 mm, length 3 mm, λ 2800 to 5000 A). The plates are afterwards evaluated (for Ca 3933·7, 3968·5; Zr 3438·2; La 3949·1, 3453·2) by non-recording microphotometry, the Seidel differences being converted into concn. of Ca and Zr by the use of standard calibration curves prepared from admixtures with Bi_2O_3. The coeff. of variation for single spectra is $\pm$ 5% for Zr and $\pm$ 8·5% for Ca at concn. of $\simeq$ 300 p.p.m. W. J. BAKER

3279. **Separation of zinc and cobalt.** Yu. V. Morachevskiĭ and Z. S. Bashun (Inst. of Chem. of Silicates, Acad. Sci., USSR). *Zavod. Lab.*, 1958, **24** (1), 20–21.—The neutral soln. (100 ml) is poured into a stirred soln. of NaOH (50 ml of 25%) to give a clear liquid. If the soln. is cloudy more NaOH may be required. Then a 10% H_2O_2 soln. is added dropwise during stirring until pptn. of Co^{3+} is apparently complete. A soln. of $FeCl_3$ is then added so that the pptd. $Fe(OH)_3$ can act as a collector of traces of Co. A re-pptn. may be necessary. The efficiency of the separation of Co and Zn was tested radiochemically with ^{65}Zn and ^{60}Co.
G. S. SMITH

3280. **Spectrophotometric determination of zinc and other metals with $\alpha\beta\gamma\delta$-tetraphenylporphine.** R. E. Bisque and C. V. Banks (Iowa State Coll., Ames). *U.S. Atomic Energy Comm.*, Rep. ISC-781, 1957, 28 pp.—*Cf. Anal. Chem.*, 1957, **29** (4, Part I), 522–526. (*Anal. Abstr.*, 1957, **4**, 3249.)

3281. **The application of Mercupral to the photometric determination of small amounts of mercury in ores.** J. Michal, E. Pavlíková and J. Zýka (Inst. für Erzforschung, Prague). *Z. anal. Chem.*, 1958, **159** (5), 321–327.—The intense yellow - brown complex formed by the reaction between tetraethylthiuram disulphide (Dicupral) and Cu^{2+} itself reacts with Hg^{2+} and Ag^+ to form a stable, although colourless, complex (*cf.* Michal and Zýka, *Anal. Abstr.*, 1957, **4**, 2121). The diminution of colour in this reaction is the basis of an indirect photometric determination of Hg in inorganic materials. The reagent consists of the separated Dicupral - Cu complex (Mercupral) in benzene soln. Prior separation of Hg from most of the usual elements is achieved by distillation of Hg from an HCl - HNO_3 soln. of the sample. After extraction of Hg in the distillate with the reagent, the absorption is read at 420 to 430 mμ (violet or blue filter) and the Hg determined from the calibration curve. As little as 0·001% of Hg may be determined.
D. F. PHILLIPS

3282. **The analysis of thorium - boron and uranium - boron alloys.** G. A. Barnett and G. W. C. Milner (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 2307, 1958, 14 pp.—Fairly rapid procedures are described for the analysis of Th - B alloys containing up to 20% of B and U - B alloys containing $\not>$ 10% of B. Dissolution of the Th - B alloy (1 g) is effected by heating it under reflux for a few hours with conc. HCl (30 ml) and then centrifuging the cool soln. The liquid layer is decanted and the residue is washed with acidified 5% (w/v) NH_4Cl and again centrifuged, the washings being added to the decanted liquid, which is then made up to 250 ml (soln. *A*). The residue is heated with conc. HNO_3 (3 ml) plus Na_2SiF_6 (2 mg), followed by evaporation to fuming with 60% $HClO_4$ and dilution of the cool soln. with H_2O (soln. *B*). The determination of Th in an aliquot of soln. *A* and the whole of soln. *B* is made by two titrations, at pH 2·5 to 3·5, with 0·02 *M* EDTA (disodium salt) and xylenol orange as visual indicator; the two values are combined for the total Th. The U - B alloy is dissolved by heating under reflux with dil. HNO_3 and the cool soln. is made up to 250 ml with H_2O. The U is then determined in an aliquot of this soln. by double pptn. as ammonium diuranate, followed by ignition to U_3O_8. When determining B, the Th or U must be removed by passing an aliquot of the sample soln. (soln. *A* for Th - B alloys) through a cation-exchange column (Zeo-Karb 225, H form); the eluate, adjusted to pH 7, is then titrated by the mannitol - NaOH method for B. Provided that the ratio of Th (or U) to B in the sample soln. is $\not>\simeq$ 100, the max. percentage error is $\pm$ 2 for B, $\simeq$ + 0·1 for Th and $\simeq \pm$ 0·5 for U; in two series of alloys containing 0·67 and 2·9% of B, mean values of 0·5 and 2·0% of B, with corresponding Th and U of 99·3 and 99%, were found. There is no loss of B during refluxing.
W. J. BAKER

3283. **Volumetric determination of aluminium in presence of iron, titanium, calcium, silicon and other impurities.** H. L. Watts (Alcoa Res. Lab., Aluminum Co. of America, East St. Louis, Ill., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 967–970.—The aluminium mineral or product is suitably decomposed and an aliquot of the resulting acid soln., containing 0·004 to 0·11 g of Al_2O_3, is treated with K oxalate soln.; the pH is adjusted to > 11·5 with NaOH. After final adjustment of pH to 10·0 with dil. HCl, KF soln. is added and the mixture is titrated with standard HCl to the first detectable change of pH. The content of Al is calculated from a given formula. Effects of **33** possible interferences are tabulated and precision data are presented: errors up to $\simeq \pm$ 1% are quoted, and the method is stated to be rapid.
D. A. PANTONY

3284. **Analysis of trace impurities. I. Analysis of impurities in purest aluminium and aluminium compounds.** O. G. Koch (Neunkircher Eisenwerk A.-G., Neunkirchen, Saar). *Mikrochim. Acta*, 1958, (1), 92–103.—Traces of many metals that occur as impurities can be extracted from an HCl soln. containing 1 g of Al by means of ammonium tetramethylenedithiocarbamate, dithizone and $CHCl_3$ at pH 3, pH 5, pH 7 and pH 9, successively, in the presence of ammonium tartrate. The combined extracts are concentrated and Be in soln. is added as an internal reference element. The soln. is then evaporated to dryness and ashed, and the metals are determined spectrographically on graphite electrodes. The appropriate lines for each element are tabulated. Calcium, Mg and Ti are among the metals that cannot be determined by this method.
G. BURGER

3285. **Oxide determinations of sintered aluminium.** F. Eisenkolb and K. Müller. *Chem. Tech., Berlin*, 1958, **10** (1), 32–33.—Methods are critically reviewed. Oxide in sintered or powdered aluminium is determined by dissolving the metal in NaOH soln. and

measuring evolved hydrogen. A suggested apparatus, accurate to ± 0·3%, is illustrated.
C. A. FINCH

3286. **Polarographic determination of indium in bearing coatings.** R. J. Manuele and J. P. Sosa (Lab. de Ensayo de la Provincia de Buenos Aires, La Plata, Argentina). *An. Asoc. Quim. Argentina*, 1957, **45**, 202–210.—The In is determined polarographically with *N* acetic acid as supporting electrolyte after separation of Pb by electrolysis. Cadmium interferes, but can be differentiated by masking In with EDTA. Mixtures of Cd and In from which Pb has been completely removed by double electrolysis can be evaluated from the normal and EDTA-masked polarograms by means of tables given. The method gives satisfactory results when applied to synthetic mixtures.
E. C. APLING

3287. **Determination of cerium(IV) by iodate and bromate.** M. K. Joshi (Dept. of Chemistry, Hindu Univ., Benares, India). *Z. anal. Chem.*, 1958, **159** (4), 283–284 (in English).—In the method described, the metal is reduced to the tervalent state by a known excess of KSCN and the excess of KSCN is subsequently titrated with standard bromate or iodate soln. Carbon tetrachloride is used as indicator with the iodate soln. and methyl orange with the bromate. Preliminary experiments indicate that moderately large amounts of Ce^{IV} and/or H_2SO_4 do not affect the determination of KSCN, and results are accurate over a wide range of cerium concn. The method has distinct advantages over that previously described in which $KMnO_4$ is used for the titration of the KSCN (*cf. Z. anal. Chem.*, 1957, **157**, 264).
S. M. MARSH

3288. **Spectroscopic studies on rare-earth compounds. II. Comparative study of the absorption spectra of the neodymium ion in aqueous solution and in crystalline salts.** P. Krumholz (Res. Lab. of Orquima S.A., Sao Paulo, Brazil). *Spectrochim. Acta*, 1958, **10** (3), 274–280.—Wavelengths and mol. extinction values of the absorption maxima and inflections are listed for the spectra (400 to 900 mμ, slit-width 0·1 mμ) of aq. soln. of neodymium perchlorate at 3°. New absorption maxima at 625·2 and 734·4 mμ are reported. The spectra resemble closely the spectrum of single crystals of $Nd(BrO_3)_3.9H_2O$ at 77° K, which is characterised by a strong line at 427·37 mμ and weak lines at 429·47, 434·09 and 434·43 mμ (Satten, *J. Chem. Phys.*, 1953, **21**, 637). The extinction indices of the chief absorption maxima of aq. soln. of neodymium perchlorate between 3° and 60° depend linearly on $(273 + T)^{-1}$. (*Cf. Anal. Abstr.*, 1958, **5**, 3568.)
W. J. BAKER

3289. **Electrophoresis. XIX. Separation and quantitative determination of rare earths in monazite sand.** Masafumi Maki (Japan Women's Univ., Takata-toyokawacho, Bunkyo-ku, Tokyo). *Japan Analyst*, 1957, **6** (12), 779–782.—The electrophoretic separation of Y, La, Ce, Pr and Nd (*cf. Anal. Abstr.*, 1957, **4**, 2143) was applied to the analysis of monazite sand. The rare-earth hydroxides, obtained from oxalates, are treated with H_2O_2 (to reduce Ce), mixed with NaCl, citric acid and aq. NH_3 soln. and submitted to electrophoresis (*loc. cit.*). Two sections of the electropherogram containing, respectively, Y, Nd and Pr, and La and Ce, are extracted and titrated with EDTA, with Eriochrome black T as indicator. The sums of Y, Nd and Pr, and of Ce and La, can be determined with an error of ± 3%.
K. SAITO

3290. **The determination of carbon-14 in reactor coolant gas.** Chemical Services Dept., Operations Branch (Windscale, England). U.K.A.E.A. Report IGO-AM/W-113, 1958, 9 pp.—The CO_2 in the coolant gas is absorbed in conc. $Ba(OH)_2$. The ppt. of $BaCO_3$ is then treated with 2 *M* acetic acid and, after purification, the activity of the evolved CO_2 is recorded in a β-proportional counter, which is standardised by using CO_2 prepared from $BaCO_3$ of known specific activity.
G. J. HUNTER

3291. **Contributions to the identification of hydrosilicates in lime sand.** J. Petrovič (Dept. of Inorg. Chem., Acad. Sci., Bratislava, Czechoslovakia). *Chem. Zvesti*, 1958, **12** (1), 54–59.—Mixtures of calcium hydrosilicates ($CaO:SiO_2 = 0·1$, 0·2 and 0·3) were studied by means of differential thermal analysis and X-ray diffraction methods.
J. ZÝKA

3292. **Determination of germanium in ores, coals and industrial wastes.** V. A. Nazarenko, N. V. Lebedeva and R. V. Ravitskaya (Ukraine Filial of the State Inst. of Rare Metals). *Zavod. Lab.*, 1958, **24** (1), 9–13.—The method, suitable for determining ≤ 0·5 μg of Ge in 1 g of sample, is based on the extraction of Ge with CCl_4 from a 9 *N* HCl soln., followed by re-extraction with water and colorimetric determination with phenylfluorone. The initial attack depends on the nature of the sample. With coal and coke, 1 g is mixed with 0·5 g of CaO in a platinum dish and then with 6 ml of saturated $Ca(NO_3)_2$ soln. After evaporation of the water, the dish is heated at 400° to 450° to burn off most of the C, and then at 700° to 800° to give a white or brownish powder. After treatment with 5 ml of conc. HNO_3 and evaporation to dryness, 5 ml of HF is added and the evaporation is repeated. Finally 5 ml of HF and 10 ml of H_3PO_4 are added and the soln. is evaporated to remove HF, giving a syrupy liquid which is dissolved in water. With oxide and silicate ores, 0·5 to 1 g of sample is heated with 5 ml of H_3PO_4 (sp. gr. 1·7), 10 ml of 40% HF soln. and 3 to 5 ml of conc. HNO_3 to give a syrupy liquid free from HF. If the decomposition is incomplete, 5 ml of HF is added and the evaporation is repeated. The residue is extracted with hot water. Methods for decomposing resinous materials, sulphide ores, coal ash and industrial wastes are also described. The soln. (25 ml) so obtained is treated in the cold with 75 ml of 12 *N* HCl, and the cold soln. is shaken with CCl_4 (2 × 20 ml) in a separating funnel. The combined extracts are washed with 9 *N* HCl (3 or 4 × 10 ml) and then extracted with water (3 × 6 ml). The combined water extracts are mixed with 2 ml of 12 *N* HCl and 1 ml of 1% gelatin soln. and then with 1·5 ml of phenylfluorone soln. (prepared by dissolving 50 mg in 100 ml of 96% ethanol containing 0·5 ml of 6 *N* HCl), and diluted to 25 ml with water. After 30 min. the colour is compared with a series of standards, or the extinction at 530 mμ is measured. The method is free from interference by all other metals.
G. S. SMITH

3293. **Qualitative inorganic analysis. IV. A test for the detection of tin(IV).** D. Bailey, W. M. Dowson, R. Harrison and T. S. West (Coll. of Technol., Birmingham, England). *Mikrochim. Acta*, 1958, (1), 137–139 (in English).—After pptn

with the alkali-soluble sulphides of the Cu group, dissolution in 4 *N* HCl and separation from As and Sb by re-pptn. of the As and Sb with H_2S, Sn can be confirmed by the following procedure. To 4 drops of the test soln. add 4 drops of 30% tartaric acid soln. Dilute with 4 drops of H_2O and add 6 drops of a 1% soln. of N-benzoylphenylhydroxylamine in acetic acid. A white ppt. indicates Sn. Under these conditions the test is specific. At the optimum acidity of 0·6 *N* HCl, 0·03 mg of Sn can be detected in 1 ml of soln. G. BURGER

3294. **Spectrochemical analysis with a universal source unit. V. Determination of lead in high-purity zinc.** Kazuo Yasuda (Casting Res. Lab., Waseda Univ., Shinjuku-ku, Tokyo). *Japan Analyst*, 1957, **6** (12), 768–774.—To improve the sensitivity, an arc-like discharge (60 μF; 56·5 ohms; 400 μH; 700 V) between the sample and a pure carbon rod is recommended. By the use of the line pair Pb 2833·1 - Zn 2542·3, a linear working curve was obtained for 0·002 to 0·01% of Pb in zinc. The use of Pb 3683·5 or 4057·8 *vs.* Zn 4298·3 (with the sample as counter electrode) provides better sensitivity but poorer precision. The influence of the shape of the sample rod was carefully examined; no marked difference in results was observed provided that the diameter of the discharge surface was > 3 mm and the conical angle was $> 90°$. K. SAITO

3295. **Rapid determination of lead as sulphate in antifriction alloys.** E. Garate and T. Garate (Lab. Central de la R.E.N.F.E., Madrid, Spain). *Chim. Anal.*, 1958, **40** (1), 7–10.—The sample (1 g) is placed in a Pyrex-glass flask covered with a watch-glass, treated with water (10 ml), HNO_3 ($d = 1·36$) (2 ml) and HF ($d = 1·113$) (3 ml), and heated on a hot-plate at 80° to 100° till solution is complete. The mixture is boiled gently to expel nitrous fumes, then diluted with hot water; 20 ml of H_2SO_4 (1:1) is added, and the whole is diluted to 100 ml and allowed to cool. When cold, the ppt. is filtered off in a crucible with Pyrex-glass filter-plate, washed with 5% H_2SO_4 satd. with $PbSO_4$, then with ethanol, ignited at 500° to 600° and weighed. Results of 34 analyses of a sample containing Sn - Sb - Pb (10:15:75) gave a mean value of 74·96% and a standard deviation of $\pm$ 0·03. Recoveries were satisfactory from samples containing from 5 to 80% of Pb and from 80 to 5% of Sn, in the presence of 15% of Sb. Under the given conditions, amounts of Pb from 0·05 to 0·8 g were pptd. completely. Spectrographic examination showed that the $PbSO_4$ contained only traces of Sn and Sb. R. E. ESSERY

3296. **Analysis of trace impurities. II. Analysis of impurities in titanium and titanium compounds.** O. G. Koch (Neunkircher Eisenwerk A.-G., Neunkirchen, Saar). *Mikrochim. Acta*, 1958, (1), 151–158.—Traces of metal that occur as impurity can be extracted from a HCl soln. containing 1 g of Ti by means of ammonium tetramethylenedithiocarbamate (5% aq. soln.), dithizone (0·01% in $CHCl_3$) and $CHCl_3$ at pH 3, 5, 7 and 9, successively, in the presence of ammonium tartrate. The combined extracts are concentrated and Be in soln. is added as an internal reference element. The soln. is evaporated to dryness and ashed, and the metals are determined spectrographically on graphite electrodes. Quantities of impurity down to 0·00001% can be determined within $\pm$ 15%. G. BURGER

3297. **Determination of micro amounts of calcium, magnesium and aluminium in titanium metal.** Hidehiro Gotô and Shuro Takeyama (Tohoku Univ., Sendai). *Sci. Rep. Res. Insts, Tohoku Univ., A*, 1957, **9**, 138–146.—Dissolve the sample in HCl. Fuse the residue with Na_2CO_3, digest with HCl, and combine the soln. Add satd. KSCN soln. (more than 5 g per 0·5 g of Ti). Dissolve the salt that forms in H_2O. Extract the soln. with diethyl ether to remove most of the KSCN, Ti^{3+} and Fe^{2+}, and oxidise the remainder with HNO_3. Neutralise with 2 *N* NaOH, add bromine water, and precipitate the Ti and Fe with Na acetate. Determine the Ca by adding an aq. soln. of Plasmocorinth B (0·025%) and NaOH and measure the extinction at 530 mμ. To determine the Al, add $FeCl_3$ and NaOH, and filter the soln. For Mg, add to the filtrate NH_4NO_3 and oxine acetate, adjust the pH to 8·6 to 9·2 and extract with benzene. Measure the extinction of the benzene layer at 434 mμ. The concn. ranges covered were—Ca 0·03 to 0·725%, Mg 0·030 to 3·99%, and Al 0·069 to 3·05%. CHEM. ABSTR.

3298. **The photometric titration of bismuth with EDTA and its application to the determination of zirconium.** G. W. C. Milner and A. Bacon (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 2494, 1958, 18 pp.—By suitable selection of iodide concn. [0·10 to 5·0% (w/v)], milligram amounts of Bi in soln. at pH 1 to 5 can be titrated directly with EDTA (disodium salt) ($\simeq$ 0·02 *M*), the iodide end-point being determined photometrically at 450 to 460 mμ (Ilford 601 filter). Hydrolysis of Bi is suppressed by increasing the concn. of I^- in soln. The interfering ions are Cl^- and F^- (both compete with I^- for Bi, Cl^- causes turbidity), tartrate and oxalate (serious), and SO_4^{2-}, NO_3^- and ClO_4^- (small). Provided that Th is complexed with SO_4^{2-} (as Na_2SO_4), $\ll$ 2·5% of Bi in Th - Bi alloys can be determined to within 1% by direct titration as described above. This procedure can also be used for the indirect determination of milligram amounts of Zr (in 1·2 *M* $HClO_4$ soln.) by the back-titration of the excess of EDTA with 0·02 or 0·1 *M* $Bi(NO_3)_2$ soln. to the iodide end-point. Accurate results are obtained over the pH range 1 to 2 with KI concn. 0·1% (w/v), but soln. containing F^- should be titrated at pH 3 (KI concn. 20%) and the F^- preferentially complexed with Be^{2+} ($\simeq 5 \times 10^{-2}$ *M*). In the determination of small amounts of Zr in alloys containing any of the metals U, Ti, Nb, Ta, Mo, W, Pb, Fe, Cu and Sn, the Zr should be separated as insol. barium fluorozirconate, which is then dissolved in a mixture of conc. HNO_3 and H_3BO_3 (satd. soln.). After masking the F^- with Be^{2+}, the back-titration method described above can be applied to the soln. W. J. BAKER

3299. **Determination of zirconium in plutonium by ion exchange and spectrography.** R. Ko (Hanford Atomic Prod. Operation, Richland, Washington). *U.S. Atomic Energy Comm.*, Rep. HW-51831, 1957, 9 pp.—Traces of Zr (20 p.p.m. with a precision of $\pm$ 17%) in plutonium are determined by forming a nitrate complex of Pu and then using an anionic exchanger to separate the complex. The remaining soln. is analysed spectrographically for Zr, with Co as an internal standard. The sample is dissolved in HCl, cobalt ions and HF are added, the soln. is evaporated, the residue is taken up in 7·2 *N* HNO_3, and passed through Dowex 1, and the resin column is washed with 7·2 *N* HNO_3. The

combined eluate and wash liquid is evaporated, taken up in HCl, and analysed spectrographically.
CHEM. ABSTR.

3300. **Precipitation of thorium oxalate from nitric acid solutions.** A. S. Ayers (Iowa State Coll., Ames). *U.S. Atomic Energy Comm.*, Rep. TID-5223, 1952 (Declassified 1957), 405–408.—Heat a soln. (200 to 300 ml), containing $\gtrsim$ 1·5 g of Th, to boiling and add a small amount of filter-paper pulp. Add oxalic acid crystals with stirring. If the soln. is 0·2 *N* in HNO_3, 100% excess of oxalic acid is adequate; *N* HNO_3 requires 200%; 1·8 *N* requires 400% excess. Cool the soln. to room temp. and filter through a medium-speed paper. Dry at 110° in a platinum crucible and ignite at 1000°. Reproducible results were not obtained at oxalic acid concn. $>$ 0·45 *N* in the soln. after pptn.
CHEM. ABSTR.

3301. **Complexometric determination of thorium with 1-(2-pyridylazo)-2-naphthol as indicator.** A. I. Busev, L. V. Kiseleva and A. I. Cherkesov (M. V. Lomonosov Moscow State Univ.). *Zavod. Lab.*, 1958, **24** (1), 13–16.—The effects of various other ions on the determination of Th by the method of Flaschka and Abdine (*Anal. Abstr.*, 1956, **3**, 2386) are studied. The concn. of Cr^{III} must not exceed 2·5 mg per ml and that of Cr^{VI} 0·6 mg per ml. In the presence of Al the pH must be $\gtrsim$ 2·5. High concn. of U, La and Ce^{III} can be present and also low concn. of Ce^{IV}, Sn^{II} and Pb. High concn. of Fe can be present if the soln. is first treated with ascorbic acid.
G. S. SMITH

3302. **Use of organic reagents in inorganic analysis. VI. Determination of thorium and zirconium with phenylglycine o- and p-carboxylic acids.** Sachindra Kumar Datta (Chem. Lab., Darjeeling Gov. Coll., India). *Z. anal. Chem.*, 1958, **159** (4), 241–249 (in English).—Both phenylglycine-*o*-carboxylic acid (**I**) and phenylglycine-*p*-carboxylic acid (**II**) produce quant. pptn. with Th, Zr, Ti and U^{IV}. Thorium may be determined by adding a 1% soln. of **II** to the soln. which has previously been made neutral to Congo red with NaOH and heated to just below the boiling-point. After settling for 10 min., the ppt. is filtered off, washed successively with 0·1% reagent in water, hot 75% ethanol, and hot water, dried and ignited to thoria. A pH range of 3·5 to 5·2 is suitable for the determination. Silver, Hg^{I}, Sn, Fe^{III}, Ce^{IV}, Zr, Ti, citrate, phosphate, tartrate and fluoride interfere with both **I** and **II**, while Hg^{II} and Pb show interference with **I** only. The effect of small amounts of Fe^{III} may be eliminated by adding a little ascorbic acid before pptn. The procedure may be applied to the separation of thorium from cerite earths in mixtures having a thoria-earth ratio up to 1:14, and also to the extraction of Th from Indian and Brazilian monazites, results for which are compared with those obtained with the *m*-nitrobenzoic acid method. Zirconium may be determined by dissolving the material in glacial acetic acid (10 ml), diluting with water to 50 ml, heating to boiling-point, and adding a 2% aq. soln. of either **I** or **II**, followed by 1·5 ml of 2% ammonium acetate soln. After settling for 10 min., the ppt. is filtered off, washed with 80% ethanol and then hot water, dried, and ignited to ZrO_2. The presence of NH_4^+ is not harmful, but a large excess of ammonium acetate should be avoided. Other interfering ions are Ag^+, Hg^+, Sn, Fe^{2+} (very slight), Fe^{3+}, Ce^{4+}, Th, Ti, Au, phosphate, citrate, tartrate and fluoride. Mercury^{II} and Pb interfere with **I** only. The reagents cannot be applied to the separation of Th from Zr or *vice versa*, since both metals are pptd. within the same pH range. The composition of the thorium and zirconium salts formed with the reagents is briefly discussed.
S. M. MARSH

3303. **Haematein as a reagent for thorium, zirconium and uranium.** K. Srinivasulu, D. Purushottam and B. S. V. Raghava Rao (Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1958, **159** (6), 406–409 (in English).—This reagent, dissolved in acetone (62 mg per litre) and diluted to 60% acetone with water, absorbs strongly between 360 and 480 mμ. On complexing with Th, U or Zr, introduced as aq. soln. made up to 60% acetone, the peak moves from 440 mμ to between 520 and 540 mμ for Th and U, and to between 500 and 520 mμ for Zr. In 25 ml of 60% aq. acetone, 0·05 mg of Th, 0·029 mg of U_3O_8 and 0·025 mg of Zr have been determined to within $\pm$ 5%.
E. G. CUMMINS

3304. **Spot test for hydrazine.** B. R. Sant (Hindu Univ., Benares, India). *Mikrochim. Acta*, 1958, (1), 169–170.—One drop of the test soln. and 1 drop of 1% NaOH soln. are added to 1 drop of 0·02 *M* $AgNO_3$ on a spot plate and stirred. After 1 min., 1 drop of aq. NH_3 is added to dissolve the Ag_2O. Black particles of Ag denote the presence of hydrazine. The identification limit is 0·1 μg and the concn. limit is 1 in 10^6. Alternatively, the test can be carried out on filter-papertre ated with $AgNO_3$ soln. The concn. limit is then 1 in 5×10^5 and the identification limit is 0·05 μg. Other reducing substances interfere.
G. BURGER

3305. **Direct determination of the oxide content of salt-like nitrides and carbides.** R. Juza, H. Puff and H. Witt (Inst. für anorg. Chem., Univ. Kiel, Germany). *Z. anal. Chem.*, 1958, **159** (4), 277–283.—The oxide content of salt-like nitrides and carbides may be determined by dissolving the material in a 2:1 mixture of water-free methanol and water-free acetic acid, when the oxide oxygen is liberated as water. This water is determined directly by a micro Karl Fischer procedure. The reaction is carried out in an atmosphere of nitrogen and at 0° in order to prevent errors due to the generation of water by the formation of methyl acetate in the solvent. Results are tabulated for lithium and magnesium nitrides and for calcium carbide.
S. M. MARSH

3306. **Inorganic nitrogen compounds. II. The determination of sodium hyponitrate.** J. Vepřek-Šiška, F. Šmirous and V. Pliška (Inst. Inorg. Chem., High School Chem. Technol., Prague). *Chem. Listy*, 1958, **52** (1), 43–46.—*Titrimetric procedure (in the presence of nitrite)*—Add 25 ml of a soln. of the sample ($\simeq$ 0·1% w/v) in 0·001 *N* NaOH to 100 ml of 0·1 *N* $KMnO_4$ (previously made alkaline with 10 ml of 5% NaOH soln.). After 30 min. add NaOH soln. (30%) (25 ml) and Devarda's alloy, and determine the NH_3 in the usual way. *Nitrite*—Add another 25 ml of the sample soln. to 100 ml of 0·7 *N* $KMnO_4$ (made alkaline as above) and, after 20 min., add with stirring H_2SO_4 (1:10) (10 ml). After a further 20 min., decolorise the soln. with 50 ml of 0·1 *N* Na oxalate, heat to 70°, add H_2SO_4 (1:5) (10 ml) and titrate with 0·7 *N* $KMnO_4$. Make a blank determination. *Calibration curve for colorimetric determination*—Add 0·1 to 5 ml of the sample soln. (containing 150 to 200 mg of $Na_2N_2O_3$

in 100 ml) to test-tubes, make up with H_2O to 5 ml, add N NaOH (3 ml), 0·1 M $K_2[Ni(CN)_4]$ (2 ml) and heat for 20 min. on a water bath. Cool, transfer the soln. to 25-ml flasks, make up to vol. with H_2O and measure the extinction with a blue - green filter at 603 mμ. For 20 to 120 mg in 100 ml the calibration curve obeys the Beer - Lambert law. The error is ± 2·7%. The only interference, by hydroxylamine, can be avoided by carrying out the determination in 0·01 N NaOH. J. ZÝKA

3307. **Volumetric determination of nitrate ion. Simultaneous determination of nitrite.** O. R. Gottlieb and M. Taveira Magalhães (Inst. Quím. Agríc., Min. da Agric., Rio de Janeiro, Brazil). *Anal. Chem.*, 1958, **30** (5), 995–997.—Two aliquots of the sample soln. of NO_3^- are run into equal vol. of a dil. H_2SO_4 soln. of 0·08 M sulphamic acid, each soln. is made just alkaline with NaOH soln. and then evaporated to dryness. One residue is mixed with a little conc. H_2SO_4 (to bring about the reaction between HNO_3 and sulphamic acid) and then diluted with water; and the other residue is diluted first with water and then with conc. H_2SO_4 so that only HNO_2, if present, reacts. Both mixtures are titrated with standard $NaNO_2$ soln., starch - KI indicator being used externally. Nitrate and nitrite concn. are calculated from given formulae with an average error of ± 0·1% for 5 to 100 mg of KNO_3. Easily oxidised org. material, Cl^- and Co^{3+} interfere unless previously separated.
D. A. PANTONY

3308. **Rapid analysis of iron-pickling liquor. IV. Rapid determination of nitrate and nitrite in hydrofluoric - nitric acid pickling liquor.** Kimio Mukaewaki (Nippon Tokushu Steel Tube Co., Funato, Itabashi, Tokyo). *Japan Analyst*, 1957, **6** (12), 775–779.—Nitrate in an HF - HNO_3 pickling liquor is reduced with 0·2 N $FeSO_4$ in > 18 N H_2SO_4 soln. by boiling for 1 min. The excess of Fe^{2+} is titrated with 0·2 N $KMnO_4$ in the presence of H_3PO_4 (> 2·5 ml for 0·5 ml of sample). The presence of < 20 mg of Fe, < 7 mg of Cr and < 2 g of H_3BO_3 (masking agent for F^-) (> 0·3 g for 0·067 g of F^-) in the sample (0·5 ml) does not cause interference. The results are corrected for NO_2^- content. Nitrite in the sample (5 ml) is titrated in the presence of 0·2 N H_2SO_4 with 0·1 N $KMnO_4$, until the violet colour persists; the soln. is then heated to 40° and the titration is continued until the colour remains constant for 1 min. The error is < 0·2% for 3 to 12% of NO_3^-, and < 2% for ≃ 0·2% of NO_2^-.
K. SAITO

3309. **Gravimetric semi-micro and micro-determination of phosphorus as 3-oxine-12-molybdophosphate.** G. Gottschalk (Fa. Osram G.m.b.H., Ernst-Reuter-Platz, Berlin-Charlottenburg). *Z. anal. Chem.*, 1958, **159** (4), 257–271.—After a discussion of the constitution of the complex formed in the pptn. of P as 3-oxine-12-molybdophosphate and of the mechanism of the reaction, a general method for the micro- (1 to 0·05 mg) and semi-micro (10 to 0·5 mg) determination of P is described. A special apparatus is used for the pptn. (details with diagram given) and the reagent is prepared as required by mixing 42 ml of HCl (*d* 1·19), 42 ml of ammonium molybdate soln. (50 g dissolved in water to 500 ml), and 16 ml of oxine soln. (5·48 g in 6 ml of HCl diluted to 100 ml with water). The acid sample is neutralised, heated to 70° to 75°, and the reagent is added with stirring. After settling for 30 min., the ppt. is filtered off, washed with 1·6% ammonium nitrate soln. and then with water, and dried at 160° to constant weight. The method has been evaluated statistically and a relative systematic error determined, which is probably caused by solubility losses. The calculated standard deviations are ± 0·006 mg of P (n = 20) for the semi-micro procedure, and ± 3·0 μg of P (n = 24) for the micro-procedure. The accuracy of the method is not affected by the HCl concn., but is affected by that of the reacting compound. The standard deviation and systematic error are unaffected by 0·02 M Fe^{3+} (if masked with EDTA), or by unmasked 0·1 M soln. of Al or bivalent Mg, Mn, Co, Ni, Cu, Zn and Cd, 0·2 M Cl^-, 0·05 M SO_4^{2-} or ammonium nitrate.
S. M. MARSH

3310. **Investigation of bromatometric analyses. I. Determination of compounds of arsenic(III) and antimony(III) with bromate.** L. Szekeres, E. Sugár and É. Pap (Univ. Budapest, Hungary). *Z. anal. Chem.*, 1958, **159** (6), 418–422.—In the titration described, starch - iodide paste is used as a reversible indicator in the determination of H_3AsO_3 and Sb K tartrate. The error is < ± 1·0%.
E. G. CUMMINS

3311. **Qualitative analysis of antimony, bismuth and arsenic in mixtures.** V. Vukčević-Kovačević and V. Fintić (Inst. of Pharm. Chem., Univ. of Zagreb, Yugoslavia). *Croat. Chem. Acta*, 1958, **30** (1), 43–46 (in English).—The presence of Sb, Bi and As in mixtures can be shown by examination of the liquid product obtained by reaction with KI and ether in the presence of HCl. The soln. under test (4 ml) is shaken with 0·5 to 2 ml of freshly prepared satd. KI soln. and 2 to 3 ml of ether. A drop of the liquid product is then carefully placed on the bottom of a Petri dish full of water, by means of a pipette. Colour changes that can be observed at the liquid - liquid interface if one, two or all three of the elements are present are tabulated.
S. M. MARSH

3312. **Gravimetric estimation of bismuth as bismuthyl dichromate.** B. R. Sant and E. T. Varkey (Hindu Univ., Benares, India). *Z. anal. Chem.*, 1958, **159** (6), 425–427 (in English).—To a known volume of bismuth soln., aq. NH_3 was added dropwise until turbidity persisted. The cloudiness was cleared with dil. HNO_3 and the soln. heated to boiling-point. A measured excess of 0·5 M K_2CrO_4 was then added and the mixture boiled for 15 min. After being set aside for 8 to 10 hr., it was filtered through a weighed sintered glass crucible, the ppt. was washed with warm H_2O, and dried at 120° to 125°. The filtrate and washings were made up to volume and the unused chromate was determined iodimetrically with arsenous oxide. The results from the two procedures are in close agreement and show that the ppt. should be weighed as $(BiO)_2Cr_2O_7$.
E. G. CUMMINS

3313. **Determination of bismuth by selenous acid.** B. R. Sant and E. T. Varkey (Hindu Univ., Benares, India). *Z. anal. Chem.*, 1958, **159** (6), 427–429 (in English).—Bismuth is pptd. quant. as bismuth selenite by boiling an almost neutral soln. of bismuth nitrate with excess of H_2SeO_3. The excess of H_2SeO_3 is determined iodimetrically by thiosulphate or arsenous oxide soln. Alternatively, the pptd. selenite may be dried at 105° to 110° and weighed as $Bi_2(SeO_3)_2$.
E. G. CUMMINS

3314. **The photometric determination of bismuth in tin.** M. I. Shvaĭger, V. P. Paklina and A. S. Medvedeva (Magnitogorsk Ind. Tech. Inst.). *Zavod. Lab.*, 1958, **24** (1), 16–17.—The sample (1 g) is dissolved in 5 ml of aqua regia, the cooled soln. is treated with 15 ml of dil. HNO_3 (2:13) and 10 ml of 10% thiourea soln., and then with water to give a volume of 50 ml, and the extinction is measured after 15 min. with a blue filter. Calibration is based on the use of a standard containing known amounts of Bi. Small amounts of Cu, Sb and Fe do not interfere. The suitability of the method for determining from 0·04 to 0·1% of Bi in tin is confirmed. G. S. SMITH

3315. **The semi-quantitative determination of traces of sodium, potassium and magnesium in bismuth and bismuth - uranium alloys.** R. J. Webb (U.K.A.E.A., Chem. Div., Woolwich Outstation, England). A.E.R.E. Report C/R 2116, 1958, 7 pp.—The sample (3 g) is converted into oxide by solution in HNO_3, evaporation to dryness and ignition at 600°. A charge of 100 mg is placed in each of three cup-electrodes, excited in a d.c. arc at 8 amp. with a gap of 4 mm, and photographed by using a medium quartz spectrograph, which allows Mg at 280 mμ and alkali metals at 600 to 700 mμ to be recorded at the same exposure. Evaluation is by visual comparison in a spectrum comparator with a standard plate of triplicate exposures of a range of standards. The electrodes are prepared by pre-burning for 15 sec. to remove surface contamination, and background is reduced by limiting the exposure to the first 45 sec. of the burn, which ensures the recording of the total emission of Na and K and one-half that of Mg. On each sample plate, triplicate exposures of a standard, corresponding approx. to the sample in composition, are recorded, to check the response of the sample plate with that of the standard plate. Suitable lines are tabulated for the three elements, allowing for a range of 2 to 100 p.p.m., the limit of visual detection being from 1 to 10 p.p.m., according to element and line. By taking specified precautions, residual blanks were reduced to 1 to 2 p.p.m. The presence of 5% of U in the bismuth had no effect on the recorded spectra other than giving a slightly heavier background, so that synthetic standards made up from Bi can be used for Bi - U alloys of this type. R. E. ESSERY

3316. **Photonometric determination of vanadium and chromium.** C. E. Bricker and S. S. Schonberg (Princeton Univ., Princeton, N.J., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 922–928.—In photonometric titration, which resembles coulometry, the reactive species is generated in the soln. by photolysis, and measurement is made of the time and intensity of the radiation. Quinquevalent V and Cr^{VI} can be determined individually or in a mixture by dissolving the sample in a soln. of Na oxalate, $Fe_2(SO_4)_3$-$(NH_4)_2SO_4.24H_2O$ and H_2SO_4 and irradiating with a standardised mercury lamp. Ferric iron, converted into the ferrous state, reduces the V and Cr, and the rate of reduction is followed by recording the absorbance of the soln. at various time intervals. Vanadium is determined at 750 mμ and Cr at 575 mμ. EDTA (disodium salt), ammonium molybdate, ammonium persulphate and phosphoric acid interfere significantly. K. A. PROCTOR

3317. **Determination of vanadium in pure aluminium.** L. Erdey and F. Szabadváry (Inst. Gen. Chem., Tech. Univ., Budapest, Hungary). *Z. anal. Chem.*, 1958, **159** (6), 429–434.—The aluminium (1 g) (containing 0·004 to 0·05% of V) is dissolved in 25 ml of dil. HCl (1:1). If the soln. is clear, it is evaporated to dryness and the residue is dissolved in 20 ml of acetate buffer (pH 3·0). If the soln. is turbid, owing to a high silicon content, a second evaporation with HCl (1:1) is carried out before the dissolution in buffer soln. This buffered soln. is treated with 0·1 *N* $KMnO_4$ till pink, and cleared by the addition of oxalic acid (0·1 *N*, 1 to 2 ml). Then 6 ml of EDTA (disodium salt) soln. (50 mg per ml) is added to complex the iron present, followed by 4 ml of Variamine blue soln. (5 mg per ml). The blue colour is measured after 5 min. in a Pulfrich photometer, with a S57 filter. The photometric calibration is carried out with an aq. soln. of NH_4VO_3 diluted to contain 20 μg of V per ml. E. G. CUMMINS

3318. **Separation and spectrophotometric determination of microgram amounts of niobium.** G. R. Waterbury and C. E. Bricker (Univ. of California, Los Alamos Sci. Lab., N.M., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 1007–1009.—Small amounts (0·05 to 2%) of Nb in plutonium and uranium alloys can be determined by dissolving the sample in a 6·3 *M* H_2SO_4 - 1·6 *M* HF mixture, extracting with *iso*-butyl methyl ketone and estimating colorimetrically with quinol in H_2SO_4. For 38 analyses of soln. containing known amounts of Nb and various other metals, the average recovery was 99·9%, with a standard deviation of ± 2%. Tantalum and large amounts of Mo interfere. K. A. PROCTOR

3319. **Chemistry of protactinium. I. Solvent extraction of protactinium.** K. A. Kraus and Q. Van Winkle (Oak Ridge Nat. Lab., Tenn.). *U.S. Atomic Energy Comm.*, Rep. TID-5223, 1952 (Declassified 1957), 296–303.—Distribution coeff. were determined for the partition of Pa between di*iso*propyl ketone and acid soln.

II. A method of analysis of ore residues for protactinium. K. A. Kraus and A. Garen. *Ibid.*, 304–306.—After addition of ^{233}Pa (β emitter) as a tracer to the ore-residue sample, the latter was leached with HF. The Pa was concentrated by addition of zirconium iodate as a carrier. This was dissolved, and Po was removed on a silver strip. Purification was continued by extraction of Pa into *iso*propyl alcohol. The alcohol was evaporated directly on to a platinum plate for α- and β-counting and α-range measurement. The total β-count gave the chemical yield, and the concn. of ^{231}Pa was given by a combination of total α-count and range analysis. More than 90% of the α-activity was due to ^{231}Pa. CHEM. ABSTR.

3320. **Studies in bivalent chromium salts. III. Standardisation of chromous sulphate solution against common oxidising agents.** J. P. Tandon and R. C. Mehrotra (B. R. College, Agra Univ., India). *Z. anal. Chem.*, 1958, **159** (5), 353–355 (in English).—Chromous salts can be standardised with the help of potassium dichromate, sulphatocerate, potassium iodate, bromate and periodate soln. N-Phenylanthranilic acid, ferroin and diphenylbenzidine serve as reversible indicators for the titrations of potassium dichromate and ceric sulphate against chromous soln.

IV. Determination of persulphate, hydrogen peroxide, and dissolved oxygen in water. J. P. Tandon and R. C. Mehrotra. *Ibid.*, 1958, **159** (6), 422–425 (in English).—Chromous sulphate is used

in two ways—(*i*) by adding an excess and back-titrating with ferric solution, with KSCN as indicator; (*ii*) by mixing with an excess of ferrous solution and titrating the Fe^{3+} produced with chromous sulphate, with neutral red or KSCN as indicator. D. F. PHILLIPS
E. G. CUMMINS

3321. **Extraction and colorimetric determination of chromium with *sym.*-diphenylcarbazide.** J. A. Dean and M. L. Beverly (Dept. of Chem., Univ. of Tennessee, Knoxville, U.S.A.). *Anal. Chem.*, 1958, **30** (5), 977–979.—A soln. (5 to 15 ml) containing 0·25 to 1 μg of Cr (as $Cr_2O_7^{2-}$) per ml is diluted with water and to the soln. are added H_3PO_4 soln. and an exact vol. of *iso*butyl methyl ketone in a separating funnel. The mixture is cooled, treated with conc. HCl and shaken. The aq. phase is rejected and an exact vol. of the org. layer is treated with *sym.*-diphenylcarbazide reagent in a spectrophotometer cell. After mixing and allowing the soln. to stand, its absorption is measured at 540 mμ *vs.* a reagent blank. The chromium concn. is derived from a linear calibration curve. Variables and interferences, which are few, in this determination are discussed. A precision of $\simeq \pm 2\%$ may be deduced from the data presented. D. A. PANTONY

3322. **Extraction of chromium with trioctylphosphine oxide from acidic solutions of alkali-metal salts. Determination *in situ* as chromium-diphenylcarbazide complex.** C. K. Mann and J. C. White (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 989–992.—The effect of variables on the extraction of Cr^{VI} from aq. soln. (M H_2SO_4) into trioctylphosphine oxide dissolved in benzene is examined and a procedure for the determination of Cr by measuring the absorption of its *sym.*-diphenylcarbazide complex at 550 mμ is developed. The complex is formed by addition of the complexing agent in ethanol to the extract. An error of ± 9% at the 2 to 10-μg level in the presence of **10 g** of alkali-metal chlorides is quoted. D. A. PANTONY

3323. **Uranium separation with cacodylic acid.** R. Pietsch (Inst. anorg. und. anal. Chem., Univ. Graz, Austria). *Z. anal. Chem.*, 1958, **159** (5), 343–348.—Procedures previously worked out for the gravimetric determination of U with cacodylic acid (dimethylarsonic acid) (*Anal. Abstr.*, 1958, **5**, 2613) also enable U to be separated from several other metals. Conditions are described for separating U from NH_4^+, Na, K, Mg, Ca, Zn, Pb, Cu, Co, Ni, Al, Cr and Fe. Sintered glass (porosity G4) is preferred to filter-paper as the filtration medium because a more efficient removal of the impurity elements by washing is possible, and the U-containing ppt. has better ignition properties. Permissible concn. of other metals with respect to U for which satisfactory recoveries have been obtained are tabulated. D. F. PHILLIPS

3324. **Estimation of uranium. II. Spectrophotometric study.** M. Nageswara Rao and B. S. V. Raghava Rao (Chem. Lab., Andhra Univ., Waltair, India). *Z. anal. Chem.*, 1958, **159** (5), 356–358 (in English).—Complexes of U with R salt (**I**) and nitroso-R salt (**II**) have been reported (*Z. anal. Chem.*, 1954, **142**, 161). 2-Naphthol-1-sulphonic acid (**III**) also forms a complex of the form UO_2X (where X is the reagent ligand). The properties of **III** are detailed and compared with those of **I** and **II**. Absorption measurements at 440 mμ showed that Beer's law was valid for complexes at pH 4·2 formed from 16·04 mg of **III** and up to 11·7 mg of UO_2^{2+} in 50 ml of total liquid. Vanadium interfered in minute quantities, Be did not interfere and Th ($\equiv>$ 32 mg of ThO_2 in 50 ml) interfered. D. F. PHILLIPS

3325. **Spectrochemical determination of uranium-235.** H. R. Mullin, H. G. Yuster and R. L. Graff. *U.S. Atomic Energy Comm.*, Rep. TID-7531, 1957, 46–62.—The possibility of analysing uranium having a ^{235}U content as low as 0·23% is indicated by igniting the sample as U_3O_8 in a high-current arc without the use of buffers, and measuring the 3rd order line optical densities from a photographic plate. CHEM. ABSTR.

3326. **Uranium-235 - uranium-238 assay on the direct-reading optical spectrograph.** T. Lee and S. A. MacIntyre (Union Carbide Nuclear Co., Oak Ridge, Tenn.). *U.S. Atomic Energy Comm.*, Rep. TID-7531, 1957, 30–35.—A 21-ft., Eagle-mounted, direct-reading optical spectrograph with a 6-in., 15,000 line per in. concave grating in the 2nd order is evaluated for the isotopic assay of ^{235}U - ^{238}U. The samples are excited in a multi-source unit of 4 hollow discharge tubes and an automatically positioned selector mirror. Duplicate analyses, including comparison standards, are made in less than 20 min. Precisions for mixtures containing 30 to 60% of ^{235}U compared favourably with work done by the mass spectrometer. Below 20% the precision drops, approaching the precision of fission counting, due to the large relative background, which could be reduced with an instrument of high resolution. Interference from ^{234}U and ^{236}U could also be removed with sufficient resolution. CHEM. ABSTR.

3327. **Polarographic determination of uranium in the presence of molybdenum.** R. C. Propst (E. I. du Pont de Nemours & Co., Aiken, S.C.). *U.S. Atomic Energy Comm.*, Rep. DP-236, 1957, 22 pp.—A method was developed for the determination of U in radioactive soln. containing U, Al, Hg and fission products in HNO_3. The sample size is chosen so that the concn. of U in the polarographic cell is approx. 5×10^{-5} M. Interference by Mo is eliminated by the use of K phthalate soln. (pH 6·0) as the supporting electrolyte. The addition of ascorbic acid to the electrolyte avoids interference from Fe^{III} and Hg^{II}. CHEM. ABSTR.

3328. **Uranium isotopic analysis with a prism-echelle spectrograph.** R. S. Murphy (Metals and Controls Corp., Attleboro, Mass.). *U.S. Atomic Energy Comm.*, Rep. TID-7531, 1957, 18–29.—An optical emission spectrographic technique for the determination of the ^{235}U content of highly enriched samples is applied to a prism-echelle spectrograph. The system uses a 2-dimensional crossed dispersion, giving greater dispersion and greater wavelength coverage than that of a medium spectrograph. The increased dispersion allows for identification and intensity measurement of ^{238}U and ^{235}U lines. The intensity ratio of $^{238}U/^{235}U$ *vs.* ^{235}U (%) on a log - log scale is linear. Quant. analysis for ^{235}U content of product-level enriched samples agreed with mass-spectrometric determinations to within 0·2% of ^{235}U at the 95% confidence limit. This accuracy is unattainable in the presence of ^{236}U. Measurement of ^{235}U is almost within the limits of emission spectrographic techniques. CHEM. ABSTR.

3329. **The isotopic determination of uranium-235 at low concentrations by means of echelle spectroscopy.** R. S. Vogel (Mallinckrodt Chem. Works, St. Louis, Mo.). *U.S. Atomic Energy Comm.*, Rep. TID-7531, 1957, 36–45.—The difficulties of photometric measurement of close line pairs having large intensity differences are overcome by using the multiple orders of an echelle spectrograph fitted with a small filter at the focal plane. In this method the choice of an internal standard line is not limited by its relative intensity, and the same line pair can be used in all concn. ranges. The relative precision of a determination at 95% confidence level is ± 4·6% and ± 6·3% for ^{235}U concn. of 1·30% and 0·711%, respectively. CHEM. ABSTR.

3330. **The gravimetric determination of uranium in uranyl nitrate solutions (after separation by ether extraction).** Chemical Services Dept., Operations Branch (Windscale, England). U.K.A.E.A. Report IGO-AM/W-116, 1958, 6 pp.—From 0·01 to 0·4 g of U per ml in soln. containing Ca, Al, Mg, F^- and other ions can be determined accurately by the procedure given. The acidity of the soln. (≡ 0·05 to 0·2 g of U) is adjusted to between 2 and 4 *M* HNO_3, and the U is then extracted into HNO_3-conditioned diethyl ether by a continuous micro-extraction method (flow rate 3 to 4 ml per min. for 25 to 35 min.). After removal of diethyl ether by evaporation, the soln. is concentrated until $UO_2(NO_3)_2.6H_2O$ crystallises, then cooled and, after addition of cellulose powder (5 g), ignited in stages to const. wt. (U_3O_8) at 900°. A correction is applied for the isotopic ratio. W. J. BAKER

3331. **Determination of uranium hexafluoride in a gas stream.** J. Greenspan and A. S. Carlson. U.S. Patent 2,797,983, date appl., 2.7.1957.—The concn. of UF_6 is determined from the rate at which a green colour advances along a bed of salicylic acid (**I**). Thus 1·5 g of **I** was packed to a depth of 4·8 cm in a glass tube of 0·343 in. diam. With a flow rate of 6 ml per min., the green colour advanced 0·5 mm per day at 0·1 mg of UF_6 per litre, and 33 cm per day at 5 mg of UF_6 per litre. CHEM. ABSTR.

3332. **Liquid - liquid extraction of uranium and plutonium from hydrochloric acid solution with tri(*isooctyl*)amine.** F. L. Moore (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 908–911.—Variables in the extraction of ^{233}U and ^{239}Pu from HCl soln. with tri*iso*octylamine are discussed. Rapid extraction of U^{VI} and Pu^{VI} takes place from, respectively, 7 *M* and 4·8 *M* HCl into tri*iso*octylamine - xylene (1:19), whereas many listed cations, including Th^{4+}, are not extracted. Potential applications of the technique are discussed. D. A. PANTONY

3333. **The micro volumetric determination of uranium and plutonium.** J. L. Bunce (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 2407, 1958, 13 pp.—The titration cell and micro-burette used are described and illustrated. Uranium in 4 *N* H_2SO_4 is reduced with liquid zinc amalgam. After separation of the aq. phase and the addition of $MnSO_4$, the titration is started with 0·05 *N* $Ce(SO_4)_2$; when most of the U has been oxidised, 1 mg of Fe^{3+} is added as ferric ammonium sulphate and the titration is continued. The end-point is determined potentiometrically by using bright platinum and calomel electrodes. The determination of Pu is similar, but is conducted in 1 to 2*N* H_2SO_4. Uranium and Pu can be simultaneously determined. The results are reproducible to ± 0·3% when titrating 1 to 4 mg of the separate elements. G. J. HUNTER

3334. **Determination of uranium in irradiated thorium.** B. E. Paige, P. Goris and J. E. Rein (Phillips Petroleum Co., Idaho Falls, Ida.). *U.S. Atomic Energy Comm.*, Rep. IDO-14411, 1957, 11 pp.—A method is given for the dissolution, separation and determination of U in neutron-irradiated thorium. The aluminium jacket, if present, is dissolved in 6 *M* HNO_3, with 0·005 *M* $Hg(NO_3)_2.1{\cdot}5H_2O$ as catalyst. After removal of the aluminium soln., the Th is dissolved in a mixture of conc. HNO_3 and 0·04 *M* HF. To an aliquot, a known amount of natural uranium is added as an internal standard, and the U is separated from the bulk of the Th by extraction with *iso*butyl methyl ketone. The total ^{233}U concn. is determined by the mass isotope dilution method. Uranium isotopic distribution can best be determined on an aliquot to which no natural uranium has been added. Data are given for typical analyses. CHEM. ABSTR.

3335. **Measuring uranium concentration in uranium - aluminium alloy by γ-counting.** P. F. Haughton, C. R. Fultz and L. E. Burkhart (Union Carbide Nuclear Co., Oak Ridge, Tenn.). *U.S. Atomic Energy Comm.*, Rep. Y-1176, 1957, 17 pp.—The ^{235}U concn. in a U - Al reactor slug is determined by measurement of the γ-activity of the entire slug in a scintillation counter with a 2 × 4-in. NaI(Tl) crystal. The limit of error is < ± 0·5%. CHEM. ABSTR.

3336. **Potentiometric determination of halides with a glass electrode by sensitisation with sodium thiosulphate.** G. Asensi Mora (Murcia Univ., Spain). *An. Real Soc. Esp. Fis. Quim., B*, 1957, **53** (11), 697–700.—Halides (Cl^- Br^- and I^-) and SCN^- are determined by titration with 0·1 *N* $AgNO_3$ in the presence of a sensitiser; $Na_2S_2O_3$ is the most satisfactory. At the equivalence point, under the influence of light, there is decomposition in the surface of the ppt. with production of H^+ and a sharp pH change. The end-point can thus be found potentiometrically, with a glass electrode. L. A. O'NEILL

3337. **Titrimetric determination of fluorine, particularly in aluminium fluoride.** L. V. Haff, C. P. Butler and J. D. Bisso (Gen. Chem. Div., Allied Chem. & Dye Corp., Morristown, N.J., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 984–989.—The AlF_3 sample is heated at 1125° in a current of superheated steam and the distillate is collected under pure water. The resulting HF soln. is titrated with standard NaOH soln. Results are based on calibration with pure AlF_3, since recovery of F^- is not quantitative. Variables in the determination are discussed: agreement between duplicates is said to be ± 0·1% and the determination can be completed within 15 minutes. D. A. PANTONY

3338. **Amperometric titration of fluoride with thorium using a rotating palladium electrode.** W. E. Harris (Dept. of Chem., Univ. of Alberta, Edmonton, Canada). *Anal. Chem.*, 1958, **30** (5), 1000–1003.—Variables in the amperometric titration of F^- with standard Th^{4+} are discussed and the following procedure is recommended. The neutral sample soln., containing 1 to 2 μg of F^- per ml, is treated with a chloroacetic acid - K_2SO_4 - KBr - NaOH

buffer mixed with Aerosol 1B (a dibutyl sodium sulphosuccinate) and titrated with standard $Th(ClO_4)_4$ soln., a rotating palladium electrode in the presence of a ferric indicator being used. After correction of diffusion current for dilution, the end-point is found graphically. Phosphate and Al^{3+} interfere, but "moderate amounts" of Cl^-, NO_3^-, SO_4^{2-}, ClO_4^-, $B_4O_7^{2-}$, Ca^{2+} and Mg^{2+} can be tolerated. A coeff. of variation (16 replicates) of ± 1·8% at the 204-μg of F^- level and ± 20% at the 30-μg level is quoted. D. A. PANTONY

3339. **Detection of perchlorate in spot-test analysis.** F. Feigl and D. Goldstein (Lab. da Prod. Mineral, Min. da Agric., Rio de Janeiro, Brazil). *Microchem. J.*, 1958, **2** (1), 105–108.—Fusion of alkali perchlorate with $CdCl_2.2{\cdot}5H_2O$ yields Cl, which gives a blue colour with thio-Michler's ketone (4:4′-bisdimethylaminothiobenzophenone) (**I**) or a red colour with fluorescein - bromide mixture (**II**). This is superior to the test of Gooch and Kreider (*Amer. J. Sci.*, 1894, **48**, 34) in which Cl is evolved into a CO_2 atmosphere and allowed to react with starch - iodide paper. *Procedure*—Evaporate one drop of test soln. and one drop of a 2% soln. of $CdCl_2.2{\cdot}5H_2O$ in a micro test-tube (5 cm × 0·7 cm) and keep the residue at 160° for about 10 min. to remove the bound water. Then place the tube in a hole made in an asbestos plate, lay a piece of reagent paper across the mouth, and heat the bottom of the tube vigorously with a micro flame. If perchlorate is present the yellow paper acquires a blue or red circular stain almost immediately. The depth of colour is proportional to the amount of ClO_4^- present. The identification limit is 1 μg of ClO_4^- (with **I**) or 5 μg (with **II**). *Reagent papers*—Immerse S. & S. 589g paper in a 0·1% benzene soln. of **I** and dry in air. Store in the dark. Immerse S. & S. 598g paper in a very weakly alkaline soln. containing 0·1 g of fluorescein and 0·5 to 0·8 g of KBr, and dry. Moisten just before use. Should halogenate or nitrate or both be present in the test soln., fume the sample with conc. HCl. This procedure enables 1 μg of ClO_4^- to be determined in the presence of 1000 μg of NO_3^- or ClO_3^-. D. F. PHILLIPS

3340. **Use of acetylacetone extractions in ferrous analysis.** J. P. McKaveney (Univ. Pittsburgh, Pa.). *U.S. Atomic Energy Comm.*, Rep. NYO-6507, 1957, 124 pp.—*Cf. Dissert. Abstr.*, 1957, **17** (5) 971; *Anal. Abstr.*, 1958, **5**, 1218.

3341. **Complexometric determination of iron in ores and agglomerates.** N. G. Fesenko (Hydrochem. Inst., Acad. Sci., USSR). *Zavod. Lab.*, 1958, **24** (1), 25–26.—The method described by Usatenko and Mikhallova (*Anal. Abstr.*, 1956, **3**, 1360) gives results that are ≃ 5% high. The soln. should be titrated at 60° instead of at room temp. and the pH should be controlled at between 1 and 3. To determine both Fe^{3+} and Fe^{2+} in ores, the sample (0·1 g) is dissolved in dil. HCl (1:1), the soln. is diluted to 200 ml, and a 20- or 25-ml aliquot is diluted to ≃ 100 ml and neutralised to Congo red or Tropaeolin OO indicator with dil. HCl (1:10). The soln. at 60° is treated with a few crystals of sulphosalicylic acid and titrated with 0·01 *N* EDTA (disodium salt) until the violet colour disappears, to give the content of Fe^{3+}. The titrated soln. is treated at 60° to 70° with 0·1 g of $(NH_4)_2S_2O_8$ and again titrated to give the content of Fe^{2+}.
G. S. SMITH

3342. **Determination of iron, chromium and nickel by X-ray fluorescence aqueous solution method.** L. Silverman and W. W. Houk (Atomics Intern., Canoga Park, Calif.). *U.S. Atomic Energy Comm.*, Rep. NAA-SR-1788, 1957, 12 pp.—Owing to the disadvantages of the inter-element effect and the size and shape of a solid sample when analysing stainless steel by X-ray fluorescence, an aq. soln. - internal-standard method was devised. Samples were prepared by dissolving the steel in aqua regia, fuming with a mixture of HF and $HClO_4$, diluting with H_2O, and taking aliquots. The time to record 25,600 counts was measured for the K_α lines of the Fe, Cr and Ni, and for the background at 0·6 A, which was used as an internal standard. A molybdenum target X-ray tube, operated at 46 kV and 38 mA, and a LiF analysing crystal were used. The ratio of element to background was calculated, and the concn. (mg per ml) was obtained from previously prepared working curves. The limit of detection was 0·1 mg per ml, with an accuracy of 3% of the amount of the element present up to 32 mg per ml for Cr and Ni, and up to 50 mg per ml for Fe. CHEM. ABSTR.

3343. **Colorimetric determination of the iron carbide content in steel.** N. M. Popova and L. V. Zaslavskaya. *Zavod. Lab.*, 1958, **24** (1), 26–29.—The method is based on the formation, by reduction, of a brownish-red colour when the carbide obtained by anodic dissolution of steel is heated with a dilute alkaline soln. of Na picrate. An amount of the moist water-washed carbide ppt. containing between 2 and 7 mg of Fe is treated with 5 drops of 20% NaOH soln. and 25 ml of 0·1% picric acid soln., and kept at 60° to 80° for 30 min. The cooled soln. is filtered. The amount of Fe present is determined in the ppt., and the extinction of the soln. is measured with a green filter to give the carbide content.
G. S. SMITH

3344. **Photometric determination of tin in iron and steel. III. Photometric determination of tin with sodium diethyldithiocarbamate.** Hidehiro Gotô and Yachiyo Kakita (Tohoku Univ., Sendai). *Sci. Rep. Res. Insts, Tohoku Univ., A*, 1957, **9**, 253–261.—The iron and steel samples are dissolved in HNO_3. Most of the Fe is removed by co-pptn. with MnO_2, and Na diethyldithiocarbamate is added to complex the Sn. Final quantities of Fe and Mn are removed by extraction with light petroleum. Tin is then extracted by benzene and the extinction is determined with a S43 (434 mμ) filter, with benzene as the standard. A spectrophotometer at wavelength 330 mμ gives greater sensitivity. CHEM. ABSTR.

3345. **Methods for the analysis of iron and steel. Part 38. Arsenic in iron and steel.** British Standards Institution (2 Park St., London, W.1). B.S. 1121: Part 38: 1958, 6 pp.—Elementary As is pptd. by hypophosphite reduction, and determined iodimetrically. The method is applicable to iron and steel, including high chromium, high tungsten and other special highly alloyed steels, but is not applicable to steel containing Se. For arsenic contents up to 0·1% the reproducibility is ± 0·003%.
O. M. WHITTON

3346. **Volumetric estimation of cobalt by potassium ferrocyanide.** H. C. Saraswat (Dept. of Chem., Christ Church College, Kanpur). *J. Sci. Ind. Res., B, India*, 1958, **17** (1), 45–46.—Cobalt has been determined by amperometric titration against $K_4Fe(CN)_6$. C. A. SLATER

3347. **Gamma spectroscopy applied to radioactivation analysis. III. The determination of cobalt in iron using gamma-gamma coincidence measurements.** L. Salmon (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 2377 (3), 1958, 8 pp.—The method depends on the fact that the 1·16 and 1·32 MeV γ-rays from ^{60}Co are in coincidence, whereas the 1·12 and 1·30 γ-rays from ^{59}Fe are not. The equipment used for direct measurements on the irradiated standard and sample is described; briefly it consists of two sodium iodide crystal detectors connected so that the output pulses from one are recorded on a multi-channel pulse amplitude analyser only when they are coincident with pulses due to 1·32 MeV γ-rays in the second. The 12 results quoted for the analysis of B.C.S. steel samples with contents of Co from 0·017% to 0·24% are in good agreement with the B.C.S. figures, and with those obtained after radiochemical separation of the ^{60}Co from the irradiated samples. G. J. HUNTER

3348. **Detection and semi-quantitative determination of small amounts of cobalt in the presence of nickel.** I. Krausz and I. Perl (Mrs.). (Inst. for Inorg. and Anal. Chem., Loránd Eötvös Sci. Univ., Budapest). *Magyar Kém. Foly.*, 1957, **63** (12), 358.—The chromatographic analysis is carried out in a test-tube. The S. & S. 2043b paper is cut into a long, narrow strip, comprising a narrow tongue dipping into the solvent, then above a wider ($\simeq$ 8 mm) part, then a long narrow part ($\simeq$ 3 mm), where the spots are observed. On top is a wider part for suspending the paper. The solvent, which should form a layer $\simeq$ 1 cm deep, is ethanol - HCl (9 : 1), and the chromatogram is run from 15 to 20 min. at room temp. For qual. analysis, no developer is needed, because Ni has $R_F = 0·0$ to 0·1, and Co has $R_F = 0·6$. The spot due to Co is visible when more than 8 to 10 μg is present, because blue $(CoCl_4)^-$ are formed. Decolorisation occurs in air because of moisture, but on warming the paper the colour reappears. If 1 to 10 μg of Co is present, NH_4SCN (2%) is added to the solvent. Small amounts of Fe and a 1000-fold amount of Ni do not interfere. Cobalt (5 to 100 μg) is determined in the presence of Ni (5 to 1000 μg) by measuring the area of the spot, and comparing it with a series of known spots. The accuracy is $\pm$ 5 μg. Small amounts of Fe do not interfere. A. G. PETO

3349. **Spectrochemical analysis of a nickel-base high-temperature alloy.** P. V. Mohan and T. P. Schreiber (Gen. Motors Corp., Detroit 2, Mich., U.S.A.). *Appl. Spectroscopy*, 1958, **12** (1), 6–7.—In the procedure described, a single-exposure point-to-plane high-voltage spark is used. The effects of source conditions, electrode shape and atmosphere on the preburn have been studied, and reproducibility and minimum matrix effects have been obtained by the use of a nitrogen atmosphere and by a careful choice of line pairs. Analytical curves have been established for the major constituents Cr, Fe, Mo, Al and Ti, and for the minor elements Mn, Si and B. K. A. PROCTOR

3350. **Analytical chemistry of the platinum metals. VII. The microchemical detection of iridium, palladium and platinum by means of phosphonium and arsonium salts.** R. Neeb (Univ., Mainz, W. Germany). *Z. anal. Chem.*, 1958, **159** (6), 401–405.—The detection of Ir, Pd and Pt in the range 0·05 to 1·0 μg per 0·05 ml of soln. by pptn. with tetraphenylphosphonium chloride, triphenylmethylarsonium chloride and tetraphenylarsonium chloride is described. Photomicrographs are included. After reduction of Ir to Ir^{3+} with hydroxylamine hydrochloride, the detection of Pd, Pt and Os in the presence of a large excess of Ir is possible. E. G. CUMMINS

3351. **Spectrophotometric determination of rhodium and platinum in plutonium.** M. E. Smith (Los Alamos Sci. Lab., Univ. of Calif., N.M., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 912–913.—Rhodium and Pt can be determined simultaneously in the range 5 to 14 p.p.m. with a coefficient of variation of about 2%. Measurements are made at 399, 470 and 665 mμ for Pt, Rh and Pu, respectively, and correction must be made for the absorbance of the Pu at 399 and 470 mμ. Ruthenium, Pd, Os, Au and Cr interfere, but large amounts of Zr and U do not. K. A. PROCTOR

3352. **Spectrophotometric and polarographic determination of ruthenium in oxalic acid.** Š. Mesarić and M. Branica (Dept. of Phys. Chem., Inst. "Rudjer Bošković," Zagreb, Yugoslavia). *Croat. Chem. Acta*, 1958, **30** (1), 81–87 (in English).—In both the spectrophotometric and polarographic methods proposed, the ruthenium compound is converted into the volatile tetroxide, which is then absorbed in *N* oxalic acid. An intense stable yellow colour is formed within 10 min. if the soln. is heated to 80°. Examination of the soln. thus formed in a Hilger Uvispek spectrophotometer (silica cells, 10-mm path) showed that a sharp absorption band exists at 375 mμ and a broad band at 475 mμ. The extinction of the 375-mμ band against concn. of Ru is in good agreement with Beer's law over the range of concn. studied (2 to 36 μg of Ru per ml), and may be used for the determination of Ru with a relative standard error of 14% to 1%. Polarographic measurements were carried out on a pen-recording Cambridge polarograph (maximum sensitivity $2·47 \times 10^{-9}$ amp. per mm) with soln. of ruthenium tetroxide in *N* oxalic acid containing 0·006% thymol in order to suppress the polarographic maximum. The $E_{\frac{1}{2}}$ *vs.* the S.C.E. = $-$ 440 mV, and a linear regression analysis gives a regression equation $C = 3·19\,h$, where h is the height of the polarographic wave ($S = \frac{1}{2}$) in mm, and C is the concn. of Ru (μg per ml) in the soln. Over the range 4 to 120 μg per ml the relative standard errors are 15 to 0·7%. The presence of HCl, H_2SO_4 or $HClO_4$ does not affect the results, but HNO_3 or nitrites make the determination impossible. Since traces of oxygen tend to distort the residual current curve, dissolved oxygen must be removed from the soln. by passing hydrogen through it for about 15 min. before the measurement. At low concn. of Ru, the spectrophotometric method is the more accurate, but the polarographic procedure has the advantage of a wider concn. range. S. M. MARSH

3353. **Polarographic determination of ruthenium in citrate and tartrate solutions.** M. Branica and Š. Mesarić (Dept. of Phys. Chem., Inst. "Rudjer Bošković," Zagreb, Yugoslavia). *Croat. Chem. Acta*, 1958, **30** (1), 89–93 (in English).—The polarographic behaviour of Ru in the following supporting electrolytes was studied—(*a*) 0·3 *M* citric acid and 0·15 *M* NaOH, (*b*) 0·2 *M* tartaric acid and 0·1 *M* NaOH with 0·009% thymol as maximum suppressor. In the first case, the $E_{\frac{1}{2}}$ *vs.* the S.C.E. = $-$ 530 mV and, in the second case, $-$ 510 mV. In tartrate

soln. the slope of the wave is less than expected from theoretical considerations, and this might be due to irreversible reduction of ruthenium in the tartrate soln., or to imperfect resolution of two polarographic waves. The following regression equations were obtained—(*a*) $C = 3{\cdot}09\,h$ and (*b*) $C = 2{\cdot}22\,h$, where h is the height of the polarographic wave in mm ($S = \frac{1}{2}$) and C is the concn. of Ru, μg per ml. Both electrolytes are suitable for the quant. determination of Ru in the concn. range 4 to 120 μg per ml, and give relative standard errors of 1·5 to 0·8%. There is no interference from HCl, H_2SO_4 or $HClO_4$ in concn. up to 0·5 *N*, but the presence of HNO_3 or nitrites makes the determination impossible. Dissolved oxygen must be removed from the soln. before the determination by passage of hydrogen for 15 min. Greater accuracy and a wider concn. range can be obtained in citrate soln. S. M. MARSH

3354. **An extraction method for the determination of radio-ruthenium in organic samples.** D. W. Shannon (Hanford Atomic Prods. Operation, Richland, Wash.). *U.S. Atomic Energy Comm.*, Rep. HW-48736, 1957, 16 pp.—The sample is added to 3 ml of a standardised organic carrier, prepared by extracting an aq. soln. of RuNO nitrato complex with tributyl phosphate and diluting with Shell Spray Base. Upon stirring, exchange occurs between the active Ru of the sample and the Ru of the carrier. The RuNO nitrato complex is then extracted from the organic phase with 1% NaOH soln. The NaOH is neutralised with acetic acid and a standardised aq. carrier is added. The Ru is then oxidised with $HIO_4.2H_2O$ and distilled into 6*N* HCl as RuO_4. Ruthenium metal is prepared from the RuO_4 by reduction with magnesium and is weighed to give the chemical yield. The β-activity is counted to complete the determination. Reproducible results for activities as low as 35 curies per min. per ml have been obtained. CHEM. ABSTR.

3355. **Sand for making colourless glasses.** British Standards Institution (2 Park St., London). B.S. 2975: 1958, 19 pp.—The essential requirements and methods of test for sand for making colourless glasses are specified. Methods of inspection for impurities, sampling, determination of moisture content, size grading, and chemical analysis (SiO_2, Al_2O_3, total Fe, TiO_2 and Cr_2O_3) are given. O. M. WHITTON

3356. **Methods for the analysis of glass. Part 3. Recommended procedure for the analysis of potassium oxide - lead oxide - silica glasses.** British Standards Institution (2 Park St., London). B.S. 2649: Part 3, 1958, 23 pp.—Recommended procedures are described for the chemical analysis of lead crystal glass, having as its main constituents SiO_2, PbO and K_2O. Details for the determination of SiO_2, PbO, Al_2O_3, CaO, MgO, K_2O, Na_2O, total As, total Fe, TiO_2, and B_2O_3 are given. O. M. WHITTON

See also Abstracts—**3243**, Preparation of high-purity calcium oxide. **3407**, Determination of Zn in lubricating oils. **3427**, Determination of Ca. **3430**, Micro-determination of fluoride. **3433**, Determination of ^{90}Sr and ^{140}Ba. **3434**, Determination of ^{210}Po. **3562**, Apparatus for ether extraction of U. **3568**, Spectroscopy of rare earths.

3.—ORGANIC ANALYSIS

3357. **Historical developments in quantitative organic micro-analysis.** A. Steyermark (Hoffmann-La Roche Inc., Nutley, N.J., U.S.A.). *Microchem. J.*, 1958, **2** (1), 21–42.—Some developments designed to make procedures and equipment more foolproof are described. Laboratory design is discussed, with special reference to the changes that have been made to balance-tables to eliminate shocks and vibration. Modern balances of both the free-swing and damped types are briefly mentioned, as well as modern designs for furnaces, digestion units, etc. Efforts made in the U.S.A. and in the U.K. towards standardisation of equipment and methods are described, and some applications of modern techniques are given. (105 references.) D. F. PHILLIPS

3358. **New silver-containing catalysts for elemental combustion analysis.** J. Horácek and J. Körbl (Inst. of Chem., Czech. Acad. of Sci., Prague). *Chem. & Ind.*, 1958, (4), 101–102.—Addition of ammoniacal silver nitrate to soln. of $FeSO_4$, $MnSO_4$ or $Ce_2(SO_4)_3$, and heating the ppt. to 550° produces metal oxides containing finely divided Ag, which are excellent catalysts for the combustion of org. compounds at 550° in determinations of C and H. An analogous Co_3O_4 - Ag catalyst is made from a cobalt salt in the presence of NaOH. These catalysts have a high absorbing power for interfering elements, *e.g.*, S (10 to 100 mg), Cl (30 to 120 mg) and I (290 to 670 mg) per g of catalyst. Examples of analyses are given. E. J. H. BIRCH

3359. **Quantitative organic analysis. XIII. A rapid method for the micro-determination of carbon and hydrogen in organic materials.** M. Večeřa, D. Šnobl and L. Synek (VÚOS, Pardubice-Rybitví, Czechoslovakia). *Mikrochim. Acta*, 1958, (1), 9–27.—The sample is burnt as rapidly as possible in a stream of O in a quartz tube, with Co_3O_4 as catalyst. Oxides of sulphur and halogens are adsorbed by silver wool at 500°; oxides of N are adsorbed by activated MnO_2. Water and CO_2 are absorbed by $Mg(ClO_4)_2$ and NaOH - asbestos, respectively. A statistical analysis is presented which reveals the contribution of each step to the total error. The precision of the procedure, calculated from 75 analyses, is equal to that of other methods. G. BURGER

3360. **Argentimetric micro-determination of organic halogen compounds, after combustion, in the presence of Variamine blue as indicator.** L. Erdey, L. Mázor and T. Meisel (Tech. Univ., Budapest). *Mikrochim. Acta*, 1958, (1), 140–146.—The material is burnt in O in a flask and the products are absorbed in NaOH soln. containing H_2O_2. The soln. is boiled to destroy H_2O_2, then acidified with acetic acid, and urea is added to destroy nitrate. The halogen can then be titrated directly with 0·01 *N* $AgNO_3$, with Variamine blue as indicator. A white pigment, *e.g.*, TiO_2, must be added to mask the colour of the ppt. If Br is present, extra H_2O_2 is needed in the absorbing soln. If the sample contains iodine, the absorbing soln. after acidification must be reduced by the addition of metallic Mg. If S is present it can be determined by diluting the absorbing soln. with *iso*propyl alcohol and titrating the SO_4^{2-} with $Ba(ClO_4)_2$, with thoron as indicator. The *iso*propyl alcohol is boiled off, the soln. is boiled with NaOH to destroy H_2O_2, and Cl^- are determined as described above. This

method is not suitable for the determination of F. In 32 determinations on known materials the largest error was 0·2%. G. BURGER

3361. **Micro-analysis of organic fluorine compounds.** T. S. Ma (Brooklyn Coll., New York, U.S.A.). *Microchem. J.*, 1958, **2** (1), 91–104.—Micro-methods available for the analysis of fluorine compounds are reviewed; these include qualitative tests, determination of C, H, F, Cl, Br, I, S, N, O and various metals. D. F. PHILLIPS

3362. **Quantitative organic analysis. XV. Micro-determination of chlorine and bromine in organic materials.** M. Večeřa and J. Bulušek (VÚOS, Pardubice-Rybitví, Czechoslovakia). *Mikrochim. Acta*, 1958, (1), 41–51.—The sample is burnt at 750° in a stream of O in a quartz tube fitted with quartz frits. The combustion products are absorbed in H_2O_2 in a removable tube. Chloride is determined by Vieböck's method and bromide by titration with $AgNO_3$, with Brilliant yellow as indicator. For samples of known composition the ratio halogen found (%) to halogen calculated (%) has a standard deviation from 1 of ± 0·00184. For materials containing 0·05% to 2% of halogen, the halogen, after absorption in H_2O_2, can be determined colorimetrically by means of $Hg(SCN)_2$. Combustion in a flask with O and absorption in 0·01 *N* NaOH (10 ml) with a few drops of 30% H_2O_2 is a suitable method for materials of low or moderate halogen content. Fusion with K and subsequent Volhard titration affords a rapid and universal method, but the end-point is not sharp. G. BURGER

3363. **Kjeldahl determination of nitrogen. Extension to nitro and nitrogen - nitrogen single-bond compounds.** W. E. Dickinson (F. S. Royster Guano Co., Atlanta, Ga., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 992–994.—Studies showed that difficultly decomposed nitro compounds could be completely reduced by subjecting them to the high-potential Zn - HCl reduction in a non-oxidising acid before Kjeldahl digestion. Formic acid (88%) was the most satisfactory acid tested. A method derived from this gave recoveries of ≃ 99% with good precision for nitro and N–N compounds. A second method in which aluminium powder and H_2SO_4 are used at 30° before digestion gave good precision and a recovery of ≃ 99% only when applied to aromatic nitro compounds. Data for 18 compounds are listed. G. P. COOK

3364. **Determination of sulphur in organic compounds.** J. F. Alicino (Squibb Inst. for Med. Res., New Brunswick, N.J., U.S.A.). *Microchem. J.*, 1958, **2** (1), 83–90.—Prevailing and recent methods are reviewed and evaluated. The author has obtained excellent results with the oxygen-flask method of Schöniger (Regional Analytical Symposium, Philadelphia, Feb. 8, 1957) and because of its simplicity recommends it for general acceptance. With sulphanilamide and other compounds of reagent quality, all results have agreed within 0·1% of theoretical for S. Three consecutive analyses on elementary S gave 99·77, 99·72 and 99·65% on samples of 1 to 2 mg. D. F. PHILLIPS

3365. **Quantitative organic analysis. XIV. Micro-determination of sulphur.** M. Večeřa and D. Šnobl (VÚOS, Pardubice-Rybitví, Czechoslovakia). *Mikrochim. Acta*, 1958, (1), 28–40.—The sample is burnt at 700° in a stream of O in a quartz tube fitted with quartz frits. The oxides of S are adsorbed on silver wool at 450° in a removable tube. Oxides of N condense in the cool section of the combustion tube. The rate of flow of O should be between 10 and 20 ml per min. The Ag_2SO_4 is extracted with water and Ag is determined in the extract by titration with NH_4SCN. Compounds containing metals that yield thermally stable sulphates can be decomposed by a modified method in which V_2O_5 at 1200° to 1300° is used. Results are in good agreement with those obtained by Zimmermann's method. The standard deviation from the known S content of test samples, transformed to known value = 1, is ± 0·0042 calculated from 84 results. A rapid colorimetric method for the determination of 0·005% to 8% of S by conversion into K_2S and determination as H_2S by means of *p*-aminodimethylaniline is briefly described. G. BURGER

3366. **Micro-determination of sulphur in organic compounds by the silver-gauze method.** E. Bladh, K. J. Karrman and O. Andersson (Univ., Lund, Sweden). *Mikrochim. Acta*, 1958, (1), 60–67 (in English).—Oxides of S resulting from combustion of the sample in O are adsorbed on the surface of silver gauze as Ag_2SO_4, which is extracted with water and determined as Ag^+, gravimetrically or volumetrically. A systematic error arises from the fact that extractable Ag^+ arise from the heating of the gauze in the absence of S. By extracting twice only with 12-ml portions of H_2O, the loss of S compensates for the blank. Before re-use of the gauze, soluble Ag^+ must be completely extracted by treatment with 0·1 *N* KCN made approx. 0·1 *N* in Na_2CO_3. The method is suitable for samples containing > 1 mg of S, but not sufficiently precise for smaller amounts. G. BURGER

3367. **Spectrophotometric determination of carbonyl oxygen.** F. H. Lohman (Procter & Gamble Co., Cincinnati, Ohio, U.S.A.). *Anal. Chem.*, 1958, **30** (5), 972–974.—Carbonyl compounds are determined by a method based on the formation of highly coloured 2:4-dinitrophenylhydrazones. These are extracted with hexane and the absorbance of the resulting soln. is measured at 340 mμ. In this way the interference of the reagent is eliminated. Recoveries on standard known mixtures of carbonyl compounds in mineral oil in concn. of ≃ 0·03 to 0·28 mg range from 80% to 119%. K. A. PROCTOR

3368. **Spectrophotometric determination of molecular weights by use of styphnates and picrates.** V. I. Siele and J. P. Picard (Explosives Res. Section, Picatinny Arsenal, Dover, N.J., U.S.A.). *Appl. Spectroscopy*, 1958, **12** (1), 8–9.—A rapid and simple technique for the determination of the mol. wt. of a large variety of primary, secondary, tertiary, alkyl, aryl and heterocyclic amines depends on a study of the absorption of their picrate and styphnate derivatives. Beer's law is obeyed for concn. between 7 and 27 mg per litre. The error is less than ± 1%. K. A. PROCTOR

3369. **The micro-identification of organic compounds.** N. D. Cheronis (Brooklyn Coll., New York). *Microchem. J.*, 1958, **2** (1), 43–70.—A review is presented of the various methods and systematic approaches that have been used since about 1820, with special reference to the operational lower limits of the reactants which, with suitable techniques, can be identified. For most methods reviewed it is concluded that this limit is not

appreciably lower than 1 μg. No precise information is available as to the lower limits of compounds that can be identified by radiochemical methods, although it appears that this may be in the nanogram (10^{-9}) or picogram (10^{-12}) range. Recent investigations have indicated that even organic reactions that take place with appreciable speeds at 1 *M* to 0·1 *M* concn. fail to proceed below 10^{-6} *M*, thus indicating that the principles of chemical equilibria may not hold at extremely low concn. of the reacting substances. (145 references.)

D. F. PHILLIPS

3370. **Identification of organic compounds. XXI. The choice of solvent system for the paper-chromatographic separation of organic compounds.** J. Gasparič and M. Večeřa (Forschungsinst. für org. Synth., Pardubice-Rybitví, Czechoslovakia). *Mikrochim. Acta*, 1958, (1), 68–91.—The most suitable solvent system for each separation can be selected by consideration of the properties of the materials to be separated. When traces of impurities are to be separated the bulk compound should remain at the starting point. The compounds to be chromatographed should be more soluble in the stationary phase than in the mobile phase. Control of R_F values can be exerted by altering the mobile phase or the stationary phase or by converting the compounds chromatographed into derivatives with different solubility characteristics. The possibilities of, and the complications ensuing from, the use of reactive solvents are discussed. Strong acids and bases and their salts and weak acids and bases can be chromatographed in neutral aqueous solvents; for acids and bases of medium strength the pH must be controlled, and the paper should be treated with a neutral salt having the same anion as the acid in the mobile phase. The use of aqueous, non-aqueous, polar and non-polar solvents is discussed, with examples.

G. BURGER

3371. **Precise intercomparison of acids by differential potentiometric titration with hydrogen electrodes.** R. G. Bates and E. Wickers. *J. Res. Nat. Bur. Stand.*, 1957, **59** (1), 9–17.—The purity of two preparations of benzoic acid, three of oxalic acid and three of K H phthalate has been compared by a precise determination of the equivalence of each substance with the same sample of sodium carbonate. The inflection point in each titration was determined by a differential potentiometric method with the use of two hydrogen electrodes. The failure of the inflection point and the equivalence point to coincide exactly has been considered in evaluating the reliability of the procedure. When 0·06 equiv. of acid was used, the reproducibility was better than 0·002%. The method is adaptable to a variety of acids and bases and provides an additional criterion of purity.

N. E.

3372. **Improved conductimetric titration of weak bases.** W. H. McCurdy, jun., and J. Galt (Frick Chem. Lab., Princeton Univ., N.J., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 940–946.—A comprehensive study is made of the titration of weak bases, dissolved in a 1 + 1 molar mixture of dioxan and formic acid, with 0·1 *N* $HClO_4$ in a similar solvent. Provided that some reaction occurs between the base and solvent, resolution of mixtures of bases differing only by 1 pK_{BH^+} unit can be achieved. The titration must be performed at 0° if N-formyl esters are readily formed by the base. On 20 to 30-mg samples, of pK_{BH^+} 2 to 5, a precision of ± 1% is claimed.

D. A. PANTONY

3373. **Radio-assay by gas chromatography of tritium- and carbon-14-labelled compounds.** R. Wolfgang and F. S. Rowland (Sterling Chem. Lab., Yale Univ., New Haven, Conn., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 903–906.—Helium carries the material to be analysed through a chromatographic column and thermal-conductivity cell and is then converted into a counting gas by the continuous injection of methane. The mixed stream of helium and methane then passes through an internal proportional counter of special design, which detects the radio-activity of the material carried.

K. A. PROCTOR

3374. **Gas-chromatographic detection of small amounts of ether in ethylene.** J. Sverak and P. L. Reiser (Österreich. Stickstoffwerke A.-G., Linz). *Mikrochim. Acta*, 1958, (1), 159–168.—Diethyl ether and dimethyl ether in ethylene can be detected and determined in concn. as low as 0·003% and 0·005% (by vol.), respectively, by gas - liquid partition chromatography under the following conditions—column (1500 mm × 6 mm); stationary phase, sterchamol, type C22 of particle size 0·2 to 0·3 mm, carrying 20% (by wt.) of Dimethylsulfolan (2:4-dimethyltetrahydrothiophen 1:1-dioxide); column temp., 25°; carrier gas, H flowing at 1 to 1·4 ml per sec.; sample volume, 10 to 20 ml; detector, differential thermal-conductivity cells connected to a bridge circuit. A diagram of the apparatus and a drawing of the sample-introduction system are shown.

G. BURGER

3375. **Mass spectrometry of some halogen compounds.** Shun Araki and Naoharu Yamaguchi (Inst. of Techno-anal. Chem., Fac. of Engng, Tokyo Univ., Hongo). *Japan Analyst*, 1957, **6** (12), 764–767.—Neither Cl nor HCl gives reproducible mass spectrograms under ordinary conditions (electronic current 12·5 μA; ion-accelerating potential 3200 V for *m/e* 28). Both vinyl chloride (**I**) and vinylidene chloride (**II**) give distinctive peaks, the strongest corresponding to a radical produced by liberation of one Cl atom (*i.e.*, *m/e* 27 for **I** and 61 for **II**). Four computing methods involving, respectively, the use of *m/e* 96 (for **II**) and 62 (for **I**), *m/e* 26 and 27 (for both), *m/e* 96 (**II**) and 27 (**I**), and *m/e* 62 (**I**) and 61 (**II**), were applied to five mixtures and the results were in good agreement.

K. SAITO

3376. **Water determination in oil-contaminated "Freon-12" [dichlorodifluoromethane] by infra-red spectrophotometry.** W. J. Diamond (Whirlpool Corp., St. Joseph, Mich., U.S.A.). *Appl. Spectroscopy*, 1958, **12** (1), 10–11.—The oil content, which must be less than 1%, is determined at the 2·34-μ H band. The sum of water and the water equivalent value of oil (*i.e.*, water absorbed by oil) is determined at 2·67 μ. The difference between these two gives the true moisture content. The presence of 0·5 and 1·0% of oil limits the accuracy of the water determination to ± 1 and ± 2 p.p.m., respectively.

K. A. PROCTOR

3377. **Infra-red and Raman spectrum of dimethoxymethane.** J. K. Wilmshurst (Auckland Univ. Coll., N. Zealand). *Canad. J. Chem.*, 1958, **36** (1), 285–289.—The infra-red spectra of dimethoxymethane in CCl_4 (3600 to 1400 cm^{-1}) and in CS_2 (1400 to 300 cm^{-1}) have been obtained. The infra-red spectrum of the vapour, in a 10-cm cell with pressures from 2·5 to 125 mm (Hg) (3100 to 300 cm^{-1}), was also obtained, and the Raman

spectrum was better resolved than that of the liquid. The main infra-red bands are at 937, 1042, 1110, 1148, 1465, 1473 and 2997 cm^{-1}. The configuration of the molecule, in the light of data obtained from the spectra, is discussed.

P. M. SORGO

3378. **Detection of alcohols by the use of their colour reaction with vanadium oxinate.** Ichiro Kudo and Ichiro Aoki (Inst. of Polytech., Osaka City Univ., Minami-ogimachi, Kita-ku). *Japan Analyst*, 1957, **6** (12), 791–792.—The black needle-like crystals of the V^V - 8-hydroxyquinoline complex dissolve in glacial acetic acid - xylene (1:1, by vol.) (10^{-4} *M*) to give a blue soln. (max. absorption, 610 mμ) and this soln. (1 ml) gives a red coloration with alcohols (0·1 ml). Aldehydes, amines and aromatic alcohols give a yellow coloration, whilst phenols show no reaction. Steroids behave similarly; thus testosterone and oestradiol containing an aliphatic OH group give a colour reaction, whereas oesterone, containing a phenolic OH group, is unreactive. K. SAITO

3379. **New method for the determination of aldosaccharides.** J. Ek and E. Hultman (Central Lab., St. Eriks Sjukhus, Stockholm). *Scand. J. Clin. Lab. Invest.*, 1957, **9**, 315–316.—A sample of sugar in water, Na 4-aminosalicylate and glacial acetic acid (1:1:10) were mixed and heated in boiling water for 20 min., after which the colour was measured at 520 mμ. The colour was directly proportional to the sugar content. Galactose and mannose gave the same colour as glucose and corresponding extinction. Keto sugars and acetone in comparable concn. gave no colour, but acetaldehyde interfered with the reaction. NUTR. ABSTR.

3380. **Estimation of dichloroacetaldehyde.** O. P. Malhotra and V. D. Anand (Dept. of Chem., Hindu Univ., Benares, India). *Z. anal. Chem.*, 1958, **159** (4), 285–287 (in English).—Three methods have been tested and found to give fairly satisfactory results—(*a*) alkaline hydrolysis and titration of excess of alkali, (*b*) argentimetric determination of the Cl^- produced during hydrolysis, and (*c*) iodination and titration of excess of iodine. A mechanism for the hydrolysis of dichloroacetaldehyde is proposed which involves the intermediate formation of glyoxal. S. M. MARSH

3381. **Studies on the metabolism of lactaldehyde. I. Separation and determination of lactaldehyde and related 3-carbon compounds.** R. P. Sandman and O. N. Miller (Dept. of Biochem., Tulane Univ. Sch. of Med., New Orleans, La., U.S.A.). *J. Biol. Chem.*, 1958, **230** (1), 353–359.—A method is described for the separation of a mixture of lactaldehyde, acetol acetate (acetoxypropanone), lactic acid, propane-1:2-diol, and its phosphate. It involves use of two ion-exchange columns, one of Amberlite IRA-400 in the HCO_3^- form for adsorption of acidic components, and the other of Amberlite IRA-400 in the SO_3H^- form for adsorption of carbonyl compounds. The ketones are selectively eluted by hot water and the aldehydes by cold aq. Na_2SO_4. Propane-1:2-diol is not adsorbed by either column. The recoveries of the various compounds range from 92 to 106%. A micro-method for the determination of lactaldehyde is described. It is based on a modification of a standard method in which the lactaldehyde is oxidised by HIO_4 to acetaldehyde. J. N. ASHLEY

3382. **Organic chemical microscopy. III. Semicarbazones as qualitative organic derivatives of aldehydes and ketones.** R. E. Dunbar and A. E. Aaland (N. Dakota State Coll., Fargo, U.S.A.). *Microchem. J.*, 1958, **2** (1), 113–127.—Optical properties are an aid in the identification of organic compounds, especially for those having solid derivatives with melting-points too close for differentiation. Semicarbazide hydrochloride has wide applicability in the identification of aldehydes and ketones. A well-standardised procedure for the prep. of semicarbazones was used (Shriner and Fuson, "The systematic identification of organic compounds," 4th Ed., Wiley, New York, 1956). (31 photomicrographs.) D. F. PHILLIPS

3383. **Properties of hexone [*iso*butyl methyl ketone] in relation to its use for uranyl nitrate extraction.** D. Bradley (A.E.R.E., Harwell, England). A.E.R.E. Report CE/R 2408, 1958, 29 pp.—The chemical and physical properties of hexone and of possible impurities are described. Tables are given for the partition of uranyl nitrate between hexone and water, properties of H_2O - HNO_3 - hexone systems, and partition data in the H_2O - NH_4NO_3 - HNO_3 - $UO_2(NO_3)_2$ - hexone system. N. E.

3384. **The infra-red spectra of malonate esters.** R. A. Abramovitch (King's Coll., Strand, London). *Canad. J. Chem.*, 1958, **36** (1), 151–158.—The infra-red spectra of several diethyl malonate derivatives, in the form of a liquid film or liquid-paraffin mull, are examined. New compounds included are diethyl 2-cyano-1-methylethylmalonate, prepared by the action of diethyl malonate on allyl cyanide in ethanolic Na ethoxide, and diethyl ethyldeuteromalonate (b.p. 211° to 212°), made by the action of D_2O on Na diethyl ethylmalonate. Most of the compounds show a splitting of the ester carbonyl stretching band, in the ranges 1739 to 1771 cm^{-1}, and 1731 to 1761 cm^{-1}. This is not due to enolisation and conjugate chelation, or association. The reason for the splitting of the band, as well as the influence of α-substituents, is discussed. P. M. SORGO

3385. **Micro-determination of secondary aliphatic amines.** D. J. Morgan (British Nylon Spinners Ltd., Pontypool, Mon.). *Mikrochim. Acta*, 1958, (1), 104–110 (in English).—Secondary aliphatic amines from C_2 to C_6 in the range 1 to 10 micro-equiv. in primary amines can be determined from the extinction of their nitrosamines with an average error of + 4·1% by the use of an extinction coeff. of 5200. The sample is dissolved in aq. acetic acid and excess of $NaNO_2$ is added at 50°. The soln. is kept at 50° for 1 to 2 min., cooled, made strongly alkaline with NaOH and extracted with hexane. The extinction of the soln. is measured at 235 mμ. The soln. of nitrosamines conform to Beer's law over the range 5×10^{-6} to 5×10^{-4} *M*. Only one calibration curve is needed. Diethylamine can be determined with a greater error. The method is not applicable to dimethylamine. G. BURGER

3386. **Colour reaction of N-alkylhydroxylamines.** Masaichiro Masui and Sumiko Fujioka (Pharm. Fac., Osaka Univ., Toyonaka). *Japan Analyst*, 1957, **6** (12), 762–764.—N-Methyl-, N-ethyl-, N-*n*-propyl- and N-*iso*propyl-hydroxylamines undergo a redox reaction with iodine soln. (1·3% in glacial acetic acid) in an acetate buffer (pH optional) to give HNO_2, which is detected by the Griess method. The limit of detection for the first two compounds

is $\simeq$ 0·3 μg and for the remaining two is $\simeq$ 5 μg. Hydroxamic acids exhibit a similar coloration, whereas oximes and nitroparaffins show only a very faint coloration unless boiled with mineral acids. K. SAITO

3387. **Detection of organic oxidants in spot tests. Distinction between chloramine T and alkali hypochlorite.** F. Feigl and R. A. Rosell (Min. of Agric., Rio de Janeiro, Brazil). *Z. anal. Chem.*, 1958, **159** (5), 335–339.—Certain organic oxidants can be detected by spot reactions on paper impregnated with thio-Michler's ketone (4:4′-tetramethyldiaminothiobenzophenone) or tetrabase (4:4′-tetramethyldiaminodiphenylmethane). Blue stains are formed. The limit of detection lies between 0·04 and 1·3 μg. N-Bromo- or chloro-succinimide reacts with a thallium salt soln. to form brown TlO(OH) making possible a selective detection of this compound, with a detection limit of 3 μg. Chloramine T can be detected in alkali soln. in the presence of alkali hypochlorite after decomposition of hypochlorite with H_2O_2 and $ZnCl_2$, followed by treatment with thio-Michler's ketone. D. F. PHILLIPS

3388. **The estimation of maleic hydrazide by paper chromatography.** W. A. Andreae (Science Service Lab., Canada Dept. of Agric., London, Ontario). *Canad. J. Biochem. Physiol.*, 1958, **36** (1), 71–74.—Maleic hydrazide (**I**) can be determined with an error of $\simeq$ 35% in partially purified plant-tissue extracts, such as indolylacetic acid oxidase prep., without preliminary purification. The method is not applicable to plants treated with **I** because of the presence of interfering substances. *Procedure*—Apply the sample soln. (10 μl containing 0·2 to 5 μg of **I**) to a 40-cm strip of Whatman No. 1 filter-paper and develop for 18 hr. by the ascending technique with either *n*-butanol - acetic acid - H_2O (5:1:4) or *iso*propyl alcohol - aq. NH_3 - H_2O (7:1:2). Dry and spray with a freshly prepared mixture of equal vol. of 1% $FeCl_3$ and 1% $K_3Fe(CN)_6$ soln. After 10 min., measure the area of the coloured spot, which is directly proportional to the logarithm of the concn. of **I**. The R_F values of **I** with the two solvent systems are 0·59 and 0·38, respectively. A. R. ROGERS

3389. **Determination of thiols in mixtures of carbohydrates and in gas.** J. Romováček and J. Bednář (Fac. of Fuels, High School of Chem. Technol., Prague). *Paliva*, 1958, **38** (1), 9–12.—The colorimetric determination of thiols with *p*-aminodimethylaniline in the presence of $FeCl_3$ and the potentiometric and amperometric titration of thiols were compared and critically evaluated. The amperometric titration with the use of $AgNO_3$ soln. was found to be the most rapid, reproducible and sensitive. When determining a thiol in the presence of CS_2, the sample is agitated with an excess of 0·01 *N* $AgNO_3$, CS_2 dissolved in the water phase is removed with a stream of N and the excess of 0·01 N $AgNO_3$ is determined by amperometric titration with a 0·01 *N* soln. of *n*-dodecanethiol in *iso*propyl alcohol. J. ZÝKA

3390. **The infra-red spectrum of some metallic chelate compounds. I. Rubeanates.** J. R. Barceló (Inst. Optics, Madrid, Spain). *Spectrochim. Acta*, 1958, **10** (3), 245–249.—The i.r. spectra between 4000 and 400 cm^{-1} for rubeanic acid (dithiooxamide) and its Cu, Ni, Co, Ag and Hg complexes are reported and discussed in relation to mol. structure. Formulae are proposed for the acid and its metal chelates in the solid state. For rubeanic acid there are three strong bands at 3304, 3205 and 3135 cm^{-1}, the spectrum resembling that of N-methylacetamide. For the Cu, Ni and Co complexes, the 3135 cm^{-1} band persists, but the other two bands are replaced by one at $\simeq$ 3230 cm^{-1}. W. J. BAKER

3391. **Infra-red spectra of silyl and silyl-d_3 cyanides.** H. R. Linton and E. R. Nixon (The Univ., Philadelphia, Pa., U.S.A.). *Spectrochim. Acta*, 1958, **10** (3), 299–306.—The spectra of gaseous SiH_3CN (**I**) and SiD_3CN (**II**) at 28° are reported, with the vibrational assignments and structural interpretations. For **I**, the chief absorption bands are at $\simeq$ 600, 920, 1360 and 2200 cm^{-1}, whilst for **II** they are at $\simeq$ 600, 700, 1080, 1600 and 2210 cm^{-1}. There is very strong evidence for a normal cyanide rather than *iso*cyanide structure for **II**. W. J. BAKER

3392. **New methods in gas analysis. IV. Vapour pressure of phenol by microchemical method.** E. Schulek, E. Pungor and J. Trompler (L. Eötvös Univ., Budapest). *Mikrochim. Acta*, 1958, (1), 52–59.—Modifications to a previously described apparatus (Schulek *et al.*, *Ibid.*, 1956, 1005; 1957, 85) for the analysis of the vapour over a liquid enable it to be used for the vapour of phenol over pure phenol or over phenol - water mixtures between 50° and 70°. A measured sample from the vapour space is swept by a stream of air into an absorption vessel and determined by titration. Replicate determinations lie within 2·5% of the mean. The chemically determined concn. of the vapour yields significantly higher values for vapour pressure than a physical method, the difference increasing at higher temp. This difference indicates partial association of phenol in the vapour phase. G. BURGER

3393. **Gas - liquid chromatography. I. Retention-volume data of certain tar acids.** L. Irvine and T. J. Mitchell (Royal College of Sci. and Technol., George Street, Glasgow, Scotland). *J. Appl. Chem.*, 1958, **8** (1), 3–6.—Corrected retention volumes per gram of stationary phase (Apiezon L stopcock grease) are given for 30 phenols at 183°, 155° and 135°. The data were obtained on a 4-ft. column with Celite-535 as the supporting medium, nitrogen as the carrier gas and thermal conductivity detection. The increase in resolution attained as the temp. decreases is offset by broadening of the peaks to the extent that overlapping may occur. This, coupled with decrease in peak height, makes 135° less satisfactory for the detection of traces of components having retention times $<$ 70 min. K. A. PROCTOR

3394. **The functionality of phenols by bromination.** A. K. Ingberman (Bakelite Co., Bound Brook, N.J., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 1003–1004.—A soln. of Br in acetic acid, with pyridine as a catalyst, provides a specific and quant. bromination agent for the ortho and para positions of a wide variety of phenols, generally within a few minutes at room temp. Results for 25 phenols were $\not<\simeq$ 97% of theoretical. G. P. COOK

3395. **Chromatography of aromatic isomers. VIII. The separation of monohydric phenols.** J. Franc (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1958, **52** (1), 55–59.—A chromatographic method for separating monohydric phenols on paper impregnated with formamide, acetamide or dimethylformamide was

developed, and the dependence of the change of ΔK on the change of dielectric constant of the mobile and solid phase was studied. The method is suitable for the determination of phenol homologues, present as impurities (1 to 2%) in technical phenol products. Pauly's reagent was used for the detection. J. ZÝKA

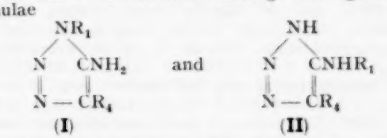
3396. **Determination of polyfunctional phenols with amidopyrine.** A. Rieche and L. Redinger (Dtsch. Akad. für Wissenschaften, Berlin-Adlershof). *Chem. Tech., Berlin*, 1958, **10** (1), 41–42.—Treatment of a polyfunctional phenol with 0·3 ml of 2% amidopyrine in 1 ml of 2 *N* aq. NH_3 and 1 ml of 2% $K_3Fe(CN)_6$ soln., and dilution to 50 ml, produces, after 2 min., a colour that is stable for 2 hr. Intensities at 428 mμ are measured and compared with suitable concn. - extinction curves for phenol, *o*- and *m*-cresol, technical *m*-cresol mixture, 2:5-xylenol, resorcinol, quinol and catechol. C. A. FINCH

3397. **Physico-chemical study of *l*-phenylacetylcarbinol and methylbenzoylcarbinol. I. Polarographic behaviour.** M. Fedoroňko (Dept. of Pharm. Chem. and Biochem., Chem. Inst., Acad. Sci., Bratislava, Czechoslovakia). *Chem. Zvesti*, 1958, **12** (1), 17–23.—The polarographic behaviour of *l*-phenylacetylcarbinol (1-hydroxy-1-phenylpropane) (**I**) and methylbenzoylcarbinol (1-benzoylethanol) (**II**) and the influence of various cations on $E_{\frac{1}{2}}$ has been studied. The shift of $E_{\frac{1}{2}}$ to positive values increases in the order $Na^+ < K^+ < Li^+ < Ca^{2+} < CH_3NH_3^+ < NH_4^+$. A two-electron reduction of both compounds on the dropping mercury electrode has been found. A soln. of pH 7 for **I** and Britton - Robinson buffer soln. for **II** are the most suitable media for polarographic analytical purposes. J. ZÝKA

3398. **Identification of organic compounds. XX. Addition products of alkylbenzyl sulphides and mercuric chloride.** M. Večeřa, J. Gasparič and M. Jureček (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Listy*, 1958, **32** (1), 144–146.—In a continuation of previous work (*cf.* Večeřa *et al.*, *Anal. Abstr.*, 1957, **4**, 1261) the addition products of alkylaryl and alkylbenzyl sulphides with mercuric chloride were prepared and purified, and their m.p., composition and constitution found. Benzyl methyl, benzyl ethyl, benzyl *n*-propyl, benzyl *iso*propyl, benzyl *n*-butyl, dibenzyl, methyl phenyl and phenyl *n*-propyl sulphides were studied. J. ZÝKA

3399. **3:4-9:10-Dibenzopyrene: fluorescence spectrum and chromatographic separation of 3:4-benzopyrene.** B. Muel, M. Hubert-Habart and Ng. Ph. Buu-Hoï (Lab. Pasteur, Inst. Radium, Paris). *J. Chim. Phys.*, 1957, **54** (6), 483–484.—The fluorescence spectrum of 3:4-9:10-[*a*,*e*]-dibenzopyrene is recorded. A chromatographic method for its separation from 3:4-benzopyrene on an alumina column and successive elutions with benzene - *cyclo*hexane (5:95), benzene - *cyclo*hexane (10:90) and pure benzene is described. The compounds are detected in the eluate by their fluorescence spectra. N. E.

3400. **The ultra-violet absorption spectra of substituted phenyl amino-1:2:3-triazoles.** E. Lieber, C. N. Ramachandra Rao and Tai Siang Chao (De Paul Univ., Chicago, Ill., U.S.A.). *Spectrochim. Acta*, 1958, **10** (3), 250–258.—The near-u.v. spectra of isomeric substituted phenyl amino-1:2:3-triazole derivatives having the general formulae



are reported and discussed in respect of the influence of R_1 (= CH_3, H, C_6H_5, $CO_2C_2H_5$, $C_6H_5CH_2$ or *p*-$NO_2C_6H_4$) and R_4 (= H, C_6H_5, $CO_2C_2H_5$) on the spectra. The quite different near-u.v. spectra of the two isomers **I** and **II** enable a qual. or quant. determination of either to be made. Isomers of type **II** are weak acids and can be determined accurately in non-aq. media (*cf.* Lieber *et al.*, *Anal. Abstr.*, 1957, **4**, 4013). The spectra between 235 and 320 mμ are also diagnostic for identifying mol. structures in this heterocyclic system. W. J. BAKER

3401. **Infra-red absorption spectra of some pyridine- and quinoline-monocarboxylic acids.** P. O. Lumme (Univ. Helsinki, Finland). *Suomen Kem., B*, 1957, **30** (11), 204–208 (in English).—The infra-red spectra of benzoic, pyridine-2-, -3- and -4-carboxylic and quinoline-2-, -6- and -8-carboxylic acids have been recorded in the wavelength range 2·5 to 15 μ. N. E.

3402. **A new method for the identification of small quantities of nicotinic acid and its esters.** D. R. Pye (County Lab. Ltd., Stanmore, Middx., England). *J. Soc. Cosmet. Chem.*, 1958, **9** (1), 62–63.—Nicotinic acid and certain of its esters react with 1-chloro-2:3-epoxypropane to give a lemon-yellow colour, 0·5 μg of nicotinic acid and 5 μg of tetrahydrofurfuryl, methyl and *n*-heptyl nicotinates being readily detected on chromatograms examined by u.v. light. With the solvent used (*n*-butanol saturated with 1·5 *N* aq. NH_3), the R_F values are very close together. H. B. HEATH

3403. **Polarography of aromatic heterocyclic compounds. V. Reduction of pyridinecarboxylic acid aldehydes.** J. Volke (Polarographic Inst., Acad. Sci., Prague). *Chem. Listy*, 1958, **52** (1), 16–24.—The heights of the irreversible reduction waves of all three isomeric aldehydes are, owing to hydration, dependent on the pH of the supporting electrolyte. The wave height in the acid range is limited by acido - base catalysed dehydration. A decrease in the height of the waves can be observed. In the middle range of pH the current has a diffusion character. Derivatives of pyridine substituted with an aldehydic group in position 2 or 4 are reduced in two waves, and pyridine-3-carboxyaldehyde in one two-electron wave. The analytical polarographic determination of these compounds can be carried out in buffered soln. (pH 6 to 7) in concn. $0{\cdot}5 \times 10^{-4}$ *M* to 3×10^{-3} *M*. Pyridine-3-carboxyaldehyde can be determined in the presence of its 2- or 4- isomer, but the determination of the 2-isomer in the presence of the 4-substituted compound, or the determination of all the isomers in admixture is not possible. Comparison with a calibration curve is necessary for the quant. determination of mixtures of the aldehydes in the presence of *iso*nicotinic or cinchomeronic acid. The use of oscillographic polarography yields similar results. J. ZÝKA

3404. **Simple photometric method for the determination of hydrazinophthalazines.** B. Wesley-Hadžiha and F. Abaffy (Anal. Dept., "Pliva" Pharm. and Chem. Works, Zagreb, Yugoslavia). *Croat. Chem. Acta*, 1958, **30** (1), 15–19 (in English).—A procedure is described for the determination of hydrazinophthalazines, which is based on the formation of a yellow azine by the action of a freshly prepared 4% soln. of *p*-dimethylaminobenzaldehyde in 2 *N* H_2SO_4; the maximum colour intensity is obtained with 0·1 ml of this soln. Standard curves are first prepared by using standard aq. soln. containing 0·4 mg per ml of 1-hydrazinophthalazine hydrochloride (**I**) and 1:4-dihydrazinophthalazine sulphate (**II**), respectively. For the determination of **I**, 0·1 ml of the reagent is added to 1 ml of the soln. containing 60 to 140 mg of **I** in 100 ml of water. After being set aside for 10 min., the mixture is diluted with water to 50 ml and the extinction is measured in a Fisher electrophotometer AC model, 2·3-cm cells, filter 425B. Comparison with the calibration curve gives the concn. of **I**. For the determination of **II**, the soln. should contain 10 to 70 mg of **II** in 100 ml of water. The procedure is similar to that described above, except that the colour is allowed to develop for 15 min., and all measurements are carried out within 5 min. after dilution. S. M. MARSH

3405. **A study of sampling of flame gases.** C. Halpern and F. W. Ruegg (Nat. Bur. of Standards, Washington, D.C., U.S.A.). *J. Res. Nat. Bur. Stand.*, 1958, **60** (1), 29–37.—The effect of sampling conditions on the measured concn. of CO, CO_2, H and H_2O in hot flame-gases is reported for water-cooled probes of 0·027 to 0·07 cm internal diameter. The probes do not quench reactions completely, nor do they preserve the original composition of the gas; small probes are most effective. Sample composition is unaffected by probe material and shape; also by rate of sampling, unless this is high, when there are fixed gradients of temp. and composition near the sampling point. The difference between apparent quenched temp. (calculated from observed gas-compositions) and flame temp. is $\simeq 500°$ when the flame is at 3050° K, but below 2250° K this difference is so small that the compositions of gas sample and flame gas are approx. equiv., *viz*, at 1600° K. More effective quenching of reactions in probes can probably be obtained by expanding the gas to low pressure or by dilution with cold He. W. J. BAKER

3406. **Refractivity intercept - density chart for the determination of total naphthenes in gasoline.** S. Groennings (Shell Development Co., Emeryville, Calif., U.S.A.). *Bull. A.S.T.M.*, 1958, (227), 64–67.—The chart permits the vol. percentage of naphthenes or paraffins in the saturated fraction (30° to 200°) of petrol to be determined with an error of $\simeq -$ 1·1% (for C_5 to C_7 saturates). Reliability and scope are compared with those of the charts of Wood (*Anal. Abstr.*, 1956, **3**, 2786) and others. W. J. BAKER

3407. **Non-aqueous titration of zinc. Rapid method for zinc in lubricating oils.** T. L. Marple, G. Matsuyama and L. W. Burdett (Res. Dept., Union Oil Co. of California, Brea, U.S.A.). *Anal. Chem.*, 1958, **30** (5), 937–940.—The sample of oil is dissolved in benzene saturated with ammonium acetate, and to the soln. is added up to 8% by vol. of methanol or ethanol. The soln. is titrated with 0·05% dithizone in benzene, the absorption of the soln. being measured at 640 mμ. Standardisation of the dithizone soln. is performed similarly, with Zn acetate as standard, and the end-point is recognised by the intersection of the two linear arms of the absorption *vs.* vol. (ml) of the titration curve. Lead and mercury interfere, but other heavy metals can be recognised by the change of shape of the titration curve. Results are compared with those of the polarographic method, and a precision of 1 to 2% on a sample containing $<$ 0·1% of Zn is claimed. D. A. PANTONY

3408. **Chromatographic analysis of coal gas.** A. Turowska and B. Jędrzejczyk (Inst. Chem. Przeróbki Węgla, Warsaw). *Gaz, Woda i Tech. Sanit.*, 1957, **31**, 266–269.—The apparatus previously described (*cf. Anal. Abstr.*, 1958, **5**, 3409) was used for complete analysis of coal gas. Lower hydrocarbons (except methane) are determined in the column filled with the zeolite, and the remaining gases, such as H, the sum N, O and CO, and methane, in a column filled with activated charcoal on which the hydrocarbons are adsorbed but not desorbed at room temp. Carbon monoxide can be partially separated from N and O in a longer column (*e.g.*, 280 cm) filled with activated charcoal. CHEM. ABSTR.

3409. **Chromatographic determination of ethylene in coal gas.** A. Turowska and B. Jędrzejczyk (Inst. Chem. Przeróbki Węgla, Warsaw). *Gaz, Woda i Tech. Sanit.*, 1957, **31**, 229–233.—The contents of ethane, ethylene and acetylene in 4-ml samples of a coal gas were determined in a chromatographic column (180 cm in length × 0·5 cm in diam.) filled with 26 g of zeolite "Alusil" (0·2 to 0·4 mm mesh). Gaseous CO_2 at the rate of 40 ml per min. and at 230 mm pressure (Hg) was used as solvent. The accuracy of the determination of ethylene was ± 0·24%. CHEM. ABSTR.

3410. **Ultra-violet spectrophotometric determination of total pyridines and quinolines in low-temperature coal-tar distillates.** Ta-Chuang Lo Chang and C. Karr, jun. (Low-Temp. Tar Lab., Bureau of Mines, Morgantown, W. Va., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 971–972.—The bases are extracted from the samples with 10% H_2SO_4 and, after the pH of this extract has been adjusted to 12 with 10% NaOH, are extracted with *iso*octane. The absorbance at 260 mμ and 316·5 mμ is compared with that of pure compounds. Large amounts of aniline interfere. Ultra-violet absorption data for $\simeq$ 25 pyridine and quinoline bases are tabulated. K. A. PROCTOR

3411. **Four methods of determination of carbon dioxide in solid fuels.** P. O. Krumin and K. Svanks (Engng Expt. Station, State Univ., Columbus, Ohio, U.S.A.). *Bull. A.S.T.M.*, 1958, (227), 51–57.—Apparatus and procedures for determining total (free + carbonate) CO_2 in coal, coke or oil-shale are compared critically. The methods are (*i*) absorption - gravimetric, (*ii*) absorption - volumetric, (*iii*) gas-volumetric, in which gases evolved by action of dil. HCl are collected over Hg and the vol. before and after subsequent absorption of CO_2 in aq. KOH soln. are measured, and (*iv*) manometric, in which the pressure change caused by carbonate decomposition in a calibrated system at const. vol. is determined. Method (*i*) has high accuracy and good reproducibility and is suitable for fuels having a wide range of CO_2 content. Method (*ii*) is satisfactory only if the total CO_2 content of the sample

is $\gtrsim \simeq 6$ mg. Method (*iii*), which is suitable for oil-shale but not for coal, necessitates a correction factor as the results are generally low; the vol. of CO_2 liberated should be $\lesssim 10$ ml. The manometric method is accurate for high and low contents of CO_2, but requires a lengthy calibration of the apparatus periodically. W. J. BAKER

3412. **The determination of a non-ionic detergent in soap mixtures.** C. B. Stuffins (Anal. Lab., J. Bibby & Sons Ltd., Gt. Howard St., Liverpool). *Soap, Perfum., Cosm.*, 1958, **31** (4), 369–370.—The non-ionic detergent is separated from soap and inorganic material by solution in dioxan, and determined gravimetrically as a molybdophosphoric acid complex. *Procedure*—Weigh 5 g of sample into a 250-ml beaker and heat it at 100° for 30 min. Cool, add dioxan (20 ml) and set the mixture aside for 2 hr. in a fume cupboard. Filter through a No. 4 sintered glass crucible into a test-tube; wash the insoluble residue with dioxan (3 × 10 ml) and transfer the bulked soln. to a 250-ml beaker. Rinse the tube with water, adding the rinsing to the beaker, and adjust the volume to about 150 ml with water. Add HCl (20% v/v) (5 ml), $BaCl_2$ (10% w/v) (5 ml) and molybdophosphoric acid (10% w/v) (5 ml), flocculating the yellow ppt. by boiling. Set aside for 18 hr., collect the ppt. on a No. 4 sintered glass crucible and wash it with a minimum of 100 ml of water. Dry at 100° for 2 hr. and weigh. Determine the percentage of detergent in the mixture from a standard graph. H. B. HEATH

3413. **Quantitative volumetric determination of fatty alcohol in fatty alcohol sulphates by the *p*-toluidine method.** J. Jerkovits (Zschimmer und Schwarz, Oberlahnstein/Rhein, Germany). *Fette, Seif., Anstrichmitt.*, 1958, **60** (1), 44–46.—The unsulphonated material in a fatty alcohol sulphate paste is determined by extracting an acidified aq. alcoholic soln. with light petroleum, removing the solvent and weighing the dried residue. For the determination of fatty alcohol, the alcohol is removed from the aq. phase by heating it in an oven at 105°, and the organic sulphate is pptd. with *p*-toluidine hydrochloride. The complex formed is extracted with ether and the soln. is added to ethanol, neutralised to cresol red, and titrated against 0·1 *N* NaOH. *p*-Toluidine hydrochloride extracted at the same time is determined argentimetrically, with K_2CrO_4 as indicator. If the mol. wt. of the fatty alcohol is not known, it can be determined by drying an aliquot of the *p*-toluidine complex soln. and weighing the residue as described by Wickbold (*Fette, Seif., Anstrichmitt.*, 1955, **57**, 164; 1952, **54**, 394). E. HAYES

3414. **Essential oils.** British Standards Institution (2 Park St., London). B.S. 2999/1 to 15, 1958, 34 pp.—Specifications (including description, apparent density, optical rotation, refractive index, ester value, acid value, solubility in ethanol, phenols, free alcohols, ketones, and sampling and size of sample, as applicable) are given for oils of cananga, celery seed, cinnamon leaf (Ceylon), cinnamon leaf (Seychelles), cubeb, lavandin, Japanese mint, neroli, parsley seed, patchouli (East Indian), patchouli (Seychelles and African), pepper, Spanish sage, spearmint, and vetiver. O. M. WHITTON

3415. **Gas chromatography and its application to perfumery materials.** G. A. P. Tuey (Res. Lab., May & Baker Ltd., Dagenham, England). *Soap, Perfum., Cosm.*, 1958, **31** (4), 353–361.—The principles of the separation of volatile mixtures into their components by gas chromatography, and its practical application to the chemistry of essential oils and related products are discussed. Several examples are quoted. H. B. HEATH

3416. **A new method for the determination of carvone and other ketones.** F. H. L. van Os and C. Scholtens. *Parfum. Cosmét. Savons*, 1958, **13** (1), 14–16.—A colorimetric method is developed for the determination of carvone in essential oils by using the ethyl ester of 3:5-dinitrobenzoic acid. *Procedure*—To 4 ml of an alcoholic soln. of carvone, containing at least 1 mg of carvone per ml, add a methanolic soln. of ethyl 3:5-dinitrobenzoate (2%, 5 ml), followed by 0·7 *N* NaOH (1 ml). After 1 min. read the absorption at 537·5 mμ, continuing the readings at 15-sec. intervals to obtain a max., against a reagent blank. Determine the carvone content from a previously prepared standard graph. Inaccuracies occur with crude essential oils, and a preliminary treatment with activated charcoal is recommended. H. B. HEATH

3417. **Chromatographic analysis of pulps utilising direct densitometry [estimation of monosaccharides].** D. F. Durso and J. C. Paulson (Buckeye Cellulose Corp., Memphis, Tenn., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 919–922.—The pulp sample is dissolved and hydrolysed with 72% H_2SO_4, successive dilutions being made as the temp. is increased. A portion of the hydrolysate is neutralised with solid $NaHCO_3$ and, after crystallisation of the resulting Na_2SO_4, the supernatant soln. is applied to the paper. Ethyl acetate - acetic acid - water (44:20:10) is used as developer, and the sugar spots are revealed with $AgNO_3$ soln. and their concn. are estimated by direct transmission densitometry. The transmission densities of the sugars are linear with concn. over the range $\simeq 1$ to 20 μg, and recoveries of added mannose or xylose are > 90%. Close agreement was reached with the results obtained by another procedure. G. P. COOK

3418. **Silicone resin content determination by piperidine extraction.** R. L. Grantham and A. G. Hastings (Res. Lab., Hughes Aircraft Co., Culver City, Calif., U.S.A.). *J. Polym. Sci.*, 1958, **27** (115), 560–561.—In this Soxhlet-type extraction for determining the resin content of silicone mouldings and laminates, the extraction with hot piperidine is complete in 1 hr. for samples $\gtrsim$ 2 mm in thickness. B. J. WALBY

3419. **An infra-red absorption method for measuring phase transitions of waxes.** J. M. Martin, jun., R. W. B. Johnston, H. J. Cannon and M. J. O'Neal (Shell Oil Co., Houston, Tex., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 1005–1006.—Many commercial waxes show abrupt changes in the i.r. spectra as the result of phase transitions. The change occurs as a splitting of the 13·88-μ methylene rocking vibration into a 13·72 to 13·92-μ doublet at the transition temp. The method is based on the plotting of a cooling curve for the molten sample, the transmittance at 13·73 μ being plotted against the fall in temp. The m.p. and transition temp. are obtained from the drop in transmittance at these points. The reproducibility was within ± 0·5° F. G. P. COOK

3420. **Determination of zinc in rubber goods.** F. Oehlmann (Dtsch. Amt. Material u. Warenprüfung, Halle, Germany). *Plaste u. Kautsch.*, 1957, **4**, 183–184.—The rubber specimen is ashed and

heated with conc. HCl. The resulting soln. is passed through an exchange column packed with Wofatit L 150. The eluate is brought to a pH of 3 or 4 with dil. aq. NH_3 soln., heated, and titrated with $K_4Fe(CN)_6$, with diphenylamine as indicator. Tests show that Fe_2O_3, TiO_2 and SiO_2 do not interfere. CHEM. ABSTR.

3421. **Analysis of explosives by non-aqueous titration.** R. D. Sarson (Olin Mathieson Chem. Corp., Mount Braddock, Pa., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 932–937.—Trinitrotoluene, dinitrotoluene, pentaerythritol tetranitrate and Hexogen (hexahydro-1:3:5-trinitro-*sym.*-triazine) are titrated as acids in *iso*butyl methyl ketone. Nitroglycerin, nitrocellulose, mononitrotoluene and NH_4NO_3 are titrated as acids in dimethylformamide. Inorganic nitrates are titrated as bases in glacial acetic acid. Trinitro-, dinitro- and mono-nitrotoluene are differentially resolved by titrations in *iso*butyl methyl ketone and dimethylformamide. Nitro compounds are separated from inorganic nitrates by extraction of the explosives mixture with *iso*butyl methyl ketone. Analysis of synthetic explosive mixtures gave results of $\simeq 100\%$ with coeff. of variation generally between $\simeq \pm 0{\cdot}2$ and $0{\cdot}4\%$. G. P. COOK

See also Abstracts—**3449**, Determination of phenols and phenolic acids in urine. **3563**, Detection of aldehydes and ketones. **3564**, Trace analysis by gas chromatography. **3565**, High-temp. gas chromatography. **3566**, Viscometer for polymer soln. **3570**, Mulling technique for samples for i.r. spectroscopy.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Blood, Bile, Urine, etc.

3422. **International survey of clinical chemistry procedures.** E. W. Rice (Dept. of Pathol., Univ. of Pittsburgh Sch. of Med., Pa., U.S.A.). *Clin. Chim. Acta*, 1958, **3** (2), 121–130.—Analytical methods for 14 blood constituents as used by selected laboratories (about 90) throughout the world are tabulated. The results show the most favoured methods to be of the "time-honoured" variety. The author finds this disturbing and has drawn up a list of more recent (American) procedures which he suggests are more appropriate. H. F. W. KIRKPATRICK

3423. **An improved technique for assay of $^{14}CO_2$ in expired air using the liquid scintillation counter.** D. S. Fredrickson and Katsuto Ono. *J. Lab. Clin. Med.*, 1958, **51** (1), 147–151.—The method described is applicable to large vol. of expired air. A methanolic soln. of methylbenzethonium chloride, in the form of the base, is used to trap the $^{14}CO_2$, and the amount is determined in an aliquot by titration. Another aliquot is diluted with a soln. of 2:5-diphenyloxazole in toluene for counting in a scintillation spectrometer. A method for the direct trapping of CO_2 expired by small animals is also given. W. H. C. SHAW

3424. **A new urine preservative.** L. Naftalin and L. R. Mitchell (County Hosp., Lincoln, England). *Clin. Chim. Acta*, 1958, **3** (2), 197–199.—A 10% (w/v) soln. of thymol in *iso*propyl alcohol (5 ml) preserves 24-hr. samples of urine in "fresh" condition for a week at room temp. The preservative does not interfere with the usual procedures required, the two exceptions found being Hay's test for bile salts and the Zimmermann reaction for 17-oxosteroids. H. F. W. KIRKPATRICK

3425. **Spectrophotometric study of suspensions of pigmented particles.** D. Keilin and E. F. Hartree (Cambridge Univ., England). *Biochim. Biophys. Acta*, 1958, **27** (1), 173–184.—A method is described by which the spectrophotometric curve of a suspension of pigmented particles can be corrected for light-scattering. From a series of curves, taken with the sample at different distances from the light detector, the curve at zero-distance can be obtained by extrapolation. For red blood cell suspensions the extrapolated curve agrees quant. with the curve of the oxyhaemoglobin solution resulting from the lysis of the red blood cells. The application of the method to cytochrome pigments, the limitations at wavelengths between 400 and 460 mμ, and theoretical aspects are discussed. W. H. C. SHAW

3426. **Polarographic determination of oxygen tension in samples of human cerebrospinal fluid.** W. W. Alberts, E. Brochmann-Hanssen, R. B. Aird and S. Winston (Calif. Univ. Med. Center, San Francisco, U.S.A.). *Arch. Biochem. Biophys.*, 1958, **74** (1), 100–105.—The construction and use of a special polarographic cell are described. Measurements on the cerebrospinal fluid from 25 neurological patients gave an average oxygen tension of 23 mm Hg (range 16 to 30). W. H. C. SHAW

3427. **Simple spectrophotometric method for the determination of calcium.** P. V. Ferro and A. B. Ham (Dade Reagents, Inc., Miami, Fla.). *Amer. J. Clin. Path.*, 1957, **28**, 208–217.—Calcium in plasma or serum was pptd. with chloranilic acid and then dissolved in EDTA (tetrasodium salt) soln. Calcium was bound by the EDTA and the resulting pink soln. of Na chloranilate was measured spectrophotometrically at 520 mμ. The colour was only slightly affected by changes in pH from 5 to 12. Duplicate determinations in the range of 4 to 40 mg per 100 ml had an error of $< 3\%$, and for the normal range of $< 2\%$. NUTR. ABSTR.

3428. **Serum copper and iron on a single sample.** B. Zak and N. Ressler (Wayne State Univ. Coll. of Med., Detroit, Mich., U.S.A.). *Clin. Chem.*, 1958, **4** (1), 43–48.—Digest the serum (3·0 ml) in a beaker on a hot-plate with 60% $HClO_4$ - HNO_3 (1:2) (10 ml) containing 40% v/v H_2SO_4 (0·1 ml) to dense white fumes. Evaporate at medium heat until H_2SO_4 fumes are barely coming off, cool and add 0·05% 1:10-phenanthroline soln. (5·0 ml) in acetate buffer (15 ml of glacial acetic acid and 70 g of Na acetate in water diluted to 1 litre) and a little solid ascorbic acid, mix, set aside for 15 min. and measure the extinction of the iron complex at 508 mμ. Rinse the beaker with 1 ml of H_2O, add to the test soln. and then add 0·035% 2:9-dimethyl-4:7-diphenyl-1:10-phenanthroline soln. (5·0 ml) in octyl or *iso*-octyl alcohol, shake and centrifuge. Measure the extinction of the copper complex in the alcohol layer at 479 mμ. Standards and blanks are treated similarly. H. F. W. KIRKPATRICK

3429. **The absorptiometric determination of lead in urine.** Chem. Services Dept., U. K. Atomic Energy Authority (U.K.A.E.A., Risley, Warrington, England). U.K.A.E.A. Report IGO-AM/W-169, 1958, 7 pp.—The sample (100 ml) is evaporated to dryness and ignited at 500° to a white solid, with ≃ 1 ml of conc. HNO_3 to complete the oxidation. The residue is dissolved in dil. HNO_3, 40% ammonium citrate soln. (7 ml) and 10% KCN soln. (5 ml) are added and the pH is adjusted to 2·8 (thymol blue). The soln. is extracted twice with a 0·006% soln. of dithizone in $CHCl_3$ to remove Bi and other elements. The pH of the aq. phase is adjusted to 8·0 (thymol blue), and the Pb is then extracted and determined spectrophotometrically at 520 mμ (1-cm cell, H503 heat filters and blue - green filters) as the dithizonate. Results are accurate for concn. of ≃ 10 μg of Pb per 100 ml, and the method is applicable to the urine of lead workers. W. J. Baker

3430. **Determination of microgram quantities of fluoride [in biological extracts].** H. M. Nielsen (Utah State Univ., Logan, U.S.A.). *Anal. Chem.*, 1958, **30** (5), 1009–1011.—The dil. soln. are passed through an anion-exchange column in its acetate form, the column is washed, and F^- are eluted with Na acetate soln. To aliquots of the eluate are added aq. Eriochrome cyanine R and an HCl soln. of ZrO^{2+}. After mixing and allowing the soln. to stand, the absorption is measured at 527·5 mμ and the F^- concn. derived from calibration curves. Common ions in biological extracts do not interfere, and a precision of ±5% on 1 to 10-μg amounts of F^- is quoted. D. A. Pantony

3431. **The determination of iodine-131 in thyroid glands.** Chem. Services Dept., Operations Branch (U.K.A.E.A., Windscale, England). U.K.A.E.A. Report IGO-AM/W-114, 1958, 8 pp.—The sample, with added NaI as carrier, is decomposed by fusion with NaOH. Organic matter is completely removed by fusion with KNO_3 and the melt is dissolved in H_2O. Iodine is liberated by the addition of $NaNO_2$ and 16 *M* HNO_3 and is then extracted into CCl_4. Dilute H_2SO_3 soln. is added to the organic phase to convert iodine into iodide, which is then extracted into the aq. phase. Excess of SO_2 is removed by heating with 2 *M* HNO_3, and AgI is then pptd. immediately by addition of 1% aq. $AgNO_3$. The slurried ppt. is transferred to a stainless-steel counting tray, dried and weighed, and its β-activity is measured with a Geiger - Müller thin end-window counter, the tray being placed 0·4 cm distant. The observed counting rate is corrected for chemical yield, and the activity of the sample is calculated by comparison with a standard source of ^{131}I. The lower limit of detection is ≃ 15 μμC (background count ≃ 7 c.p.m., counter efficiency ≃ 10%). W. J. Baker

3432. **Methods for the investigation of radio-iodine in the serum after small test doses of iodine-131.** R. J. Block, S. C. Werner and R. H. Mandl (Boyce Thompson Inst. Plant Res., Inc., Yonkers, N.Y., U.S.A.). *Arch. Biochem. Biophys.*, 1958, **73** (1), 9–19.—Two paper-chromatographic methods are described; one is suitable for separating protein-bound iodine from iodides and the other for separating iodinated thyronines and, less satisfactorily, iodinated tyrosines. A 125-μC test dose of $Na^{131}I$ and 15 to 25 ml of serum are required. Typical results on serum samples showing the different distribution patterns of ^{131}I are given. W. H. C. Shaw

3433. **Determination of strontium-90 and barium-140 in bone, dairy products, vegetation and soil.** H. L. Volchok, J. L. Kulp, W. R. Eckelmann and J. E. Gaetjen (Isotopes Inc., Westwood, N.J., U.S.A.). *Ann. N.Y. Acad. Sci.*, 1957, **71**, 293–304.—Samples of bone, cheese, milk and vegetation were ashed at 600° and dissolved in HCl. Strontium and Ba were converted first into their oxalates and then to their chlorides. Soil samples were extracted with ammonium acetate or HCl and the chlorides were similarly obtained. Both ^{90}Sr and ^{140}Ba were better measured as their radioactive daughter substances, ^{90}Y and ^{140}La. These were therefore separated at pH 5 by pptn. of their hydroxides, which were converted to the corresponding oxalates; ^{90}Y and ^{140}La were then measured in an anti-coincidence counter with a very low background count. When little of either ^{90}Sr or ^{140}Ba was present they were separated at the beginning on a cation-exchange resin column. Nutr. Abstr.

3434. **The determination of polonium-210 in urine and other biological materials.** Chem. Services Dept., U. K. Atomic Energy Authority (U.K.A.E.A., Risley, Warrington, England). U.K.A.E.A. Report IGO-AM/W-167, 1958, 7 pp.—The procedure is applicable to urine, blood and faeces. The whole sample (urine is collected into 25 ml of 16 *M* HNO_3) is digested with conc. HNO_3 plus conc. H_2SO_4, and then with 60% $HClO_4$, to destroy organic matter. The pale-straw soln. is diluted to ≃ 300 ml with H_2O, potassium tellurate soln. (10 ml = 10 mg of Te) is added, and Po is then co-pptd. with Te by reduction with an excess of sodium hypophosphite at ≃ 100°. The ppt. is dissolved in a saturated soln. of Br in HCl, and Te is then removed by pptn. with hydrazine hydrochloride. The acidity and vol. of the filtrate are adjusted and Po is deposited electrochemically on a polished silver disc at 80° during ≃ 90 min. The activity of the deposit is then measured in an α-scintillation counter against a standard source of Pu. Differences in energies, back-scatter and geometry are accounted for in the total bias of the method. The accuracy, based on 32 determinations on 500 ml of urine containing 5 or 11 d.p.m. of ^{210}Po, is represented by a bias of ≃ 30%. The method is valid up to 6×10^4 d.p.m. of ^{210}Po on the disc, and the limit of detection is 5 d.p.m. of ^{210}Po (d.p.m. = disintegrations per min.). W. J. Baker

3435. **Spectrophotometric method for the estimation of carbon monoxide haemoglobin in blood.** J. Brückner and F. B. Desmond (Med. Sch., Univ. of Otago, Dunedin, N.Z.). *Clin. Chim. Acta*, 1958, **3** (2), 173–178.—The method, described in detail, depends on readings at five wavelengths of the extinction of a 1 in 20 dilution of blood. From the readings, three quotients are obtained, and the required percentage is read directly from a graph. H. F. W. Kirkpatrick

3436. **Determination of ethanol in finger-tip quantities of blood.** L. A. Williams, R. A. Linn and B. Zak (Offices of Wayne County Med. Examiner, Detroit, Mich., U.S.A.). *Clin. Chim. Acta*, 1958, **3** (2), 169–172.—Brucine is employed to determine Cr^{VI} in the presence of Cr^{III}. *Calibration*—Standard soln. are prepared by diluting aliquots (2 to 10 ml) of a 4% (w/v) aq. soln. of ethanol to 100 ml with H_2O. Pipette 3 ml of $K_2Cr_2O_7$ reagent [0·071% (w/v) $K_2Cr_2O_7$ in 50% (v/v) H_2SO_4] into 25-ml volumetric flasks, add 0·1 ml of standard soln. to

each, stopper and heat in a water bath at 55° to 65° for 1 hr. Cool, add 10 ml of H_2O, 3 ml of brucine soln. [1% (w/v) in 10% H_2SO_4] and dilute with H_2O to the mark. Read at 425 mμ after 5 min. against a water blank. *Sample procedure*—Place 3 ml of $K_2Cr_2O_7$ reagent in the centre and 1 ml of 20% Na_2CO_3 in the outer compartment of a Conway unit. Add 0·1 ml of blood to the Na_2CO_3 soln., cover immediately and incubate at 55° to 65° for 1 hr. in a shallow water bath, remove, and cool. Transfer the $K_2Cr_2O_7$ soln. to a 25-ml flask, washing with H_2O (3 × 3 ml), add 3 ml of brucine soln., make up to the mark, mix and read as before.

H. F. W. KIRKPATRICK

3437. **Determination of morphine in blood and tissues.** J. C. Szerl, D. P. McLeod, F. Moya and D. H. McCurdy (Dalhousie Univ., Halifax, N.S., Canada). *Arch. Int. Pharmacodyn.*, 1957, **109**, 99–107.—After removal of impurities by benzene pptn., and adsorption on ion-exchange resin, morphine can be determined colorimetrically by the use of the Folin - Ciocalteu phenol reagent. As little as 3 μg of the free base in 1 g of blood or tissue, or 0·6 μg per ml of plasma, can be determined with recoveries of 80% or better. CHEM. ABSTR.

3438. **Quantitative estimation of barbiturates in body fluids by ultra-violet spectrophotometry.** R. Richterich (I Med. Univ.-Klinik, Basle, Switzerland). *Clin. Chim. Acta*, 1958, **3** (2), 183–196.—Plasma or urine (0·2 ml) is extracted with $CHCl_3$ at pH 7·4 and the $CHCl_3$ is extracted with 0·1 *N* NaOH. The u.v. absorption is measured over the range 220 to 280 mμ at pH 10 (specific plus non-specific) and at pH 2 (non-specific). The corrected readings show a typical curve with a peak at 240 mμ if barbiturates are present, the $E_{max.}$ being used for quant. evaluation. H. F. W. KIRKPATRICK

3439. **Ultra-micro determination of drugs in blood serum. II. Free sulphanilamide.** R. P. MacDonald and J. Ploompuu (Harper Hospital, Detroit, Mich., U.S.A.). *Mikrochim. Acta*, 1958, (1), 147–150 (in English).—The sample of serum (25 μl) is mixed with 10% trichloroacetic acid (500 μl) and centrifuged. The primary aryl group of the sulphonamide in the supernatant liquid is diazotised with HNO_2 and the excess of nitrite is destroyed with ammonium sulphamate. The diazotised sulphonamide is coupled with N-1-naphthylethylenediamine dihydrochloride to form a purplish-red azo dye. The extinction of the dye at 540 mμ is measured against a blank. The precision is equal to that of macro-methods. The calibration curve is prepared from standard soln. of sulphanilamide. Beer's law is followed. If the drug administered is known, its concn. can be found from the sulphanilamide concn. by means of a table of factors presented. The micro apparatus used has been described previously (Knights *et al.*, "Ultramicro methods for clinical laboratories," Grune and Stratton, New York, 1957).

G. BURGER

3440. **Enzymic determination of acetaldehyde in blood.** F. Lundquist (Univ. Inst. of Forensic Med., Copenhagen, Denmark). *Biochem. J.*, 1958, **68** (1), 172–177.—The method for determination of acetaldehyde in blood by diffusion into semicarbazide is interfered with by acetoacetate, which is partly converted into acetone during the procedure. A method is described which is based on the use of a K-requiring aldehyde dehydrogenase, which is easily prepared from commercial dry yeast. The rate of formation of reduced diphosphopyridine nucleotide from the oxidised form under specified conditions is determined spectrophotometrically, and is proportional to the concn. of aldehyde within a considerable range. The specificity is not complete because several aldehydes react under the analytical conditions, but none of these aldehydes, so far as is known, occurs in animals. The accuracy of the method at very low concn. of aldehyde is better than that obtained with other methods. The limit for detection of acetaldehyde in biological material is ≃ 0·1 μg per g. J. N. ASHLEY

3441. **Electromigration on filter-paper of uric acid from serum and synovial fluid.** F. Salteri, E. Cirla and A. Fasoli (Inst. of Clin. Med. and Med. Therapy, Univ. of Milan, Italy). *Science*, 1958, **127**, 85–86.—Paper electrophoresis with barbitone buffer (pH 8·6), ionic strength 0·05, on Whatman No. 31 extra-thick filter-paper, showed that uric acid in serum and synovial fluid has the same mobility as free uric acid, *viz*, slightly greater than serum albumin. Qualitative detection is made with $AgNO_3$ and Na lactate; quant. evaluation is obtained by elution and chemical or u.v. analysis.

H. F. W. KIRKPATRICK

3442. **Modifications of the fluorescence method of Weil - Malherbe and Bone to determine adrenaline and noradrenaline in human plasma.** J. Kägi, M. Burger and K. Giger (Univ. Zurich, Switzerland). *Arch. exp. Path. Pharmak.*, 1957, **230**, 470–478.—A mixture of 15 ml of blood and 5 ml of a soln. of 2 g of NaF and 3 g of $Na_2S_2O_3.5H_2O$ in 100 ml of H_2O was centrifuged at 0°, the supernatant fluid was mixed with an equal vol. of 0·2 *M* Na acetate, and adjusted with 0·5 *M* Na_2CO_3 to pH 8·4. The catecholamines were adsorbed on a column of 0·7 g of alumina (previously washed and moistened with 5 ml of Na acetate soln. of pH 8·4), washed with 10 ml of purest H_2O adjusted to pH 9·2, eluted with 5 ml of 0·2 *M* acetic acid, and rinsed with 5 ml of H_2O. To the eluate was added 2 ml of a mixture (1·3 : 1) of 4 *M* NH_4Cl and ethylenediamine hydrate (76%), the mixture was heated for 20 min. at 63°, cooled, shaken after addition of 4 g of NaCl with 8 ml of *iso*butyl alcohol for 4 min. and centrifuged at 0°. The fluorescence was determined in a cuvette (10 mm × 20 mm × 45 mm) of low fluorescence of its own. For comparison were used 10^{-10} mole of adrenaline or noradrenaline in 10 ml of 0·1 *M* acetic acid and, as blanks, the reagents employed. Precautions concerning purity of reagents are enumerated. Since light destroys the noradrenaline, the light source must not be too strong and the reading done rapidly. Interfering substances are excluded: hydroxytryptamine is not adsorbed on alumina; dihydroxyphenylalanine gives a fluorescent substance that is not extracted with *iso*butyl alcohol; *iso*propylnoradrenaline occurs only in traces in the adrenal medulla. CHEM. ABSTR.

3443. **Spectrophotometric analysis of plasma bilirubin.** R. Bourrillon (Biochem. Lab., 45, rue des Saints Pères, Paris). *Bull. Soc. Chim. Biol.*, 1958, **40** (1), 111–125.—Bilirubin (**I**) shows an absorption spectrum in the region 410 to 460 mμ. **I** from normal or haemolytic sera shows a max. at 460 mμ, alone or mixed with serum. **I** in icteric sera of biliary origin gives a much less marked peak at 450 to 455 mμ. The isolated complex from these sera gives a peak at 450 mμ. Addition of serum to biliary **I** shifts the peak from 450 mμ to

420 mμ. A macro-molecular complex is postulated in bile that is dissociated by addition of serum.
H. F. W. KIRKPATRICK

3444. **Methods for the qualitative and quantitative determination of tetroses by two new specific colour reactions.** Z. Dische and M. R. Dische (Columbia Univ., New York, U.S.A.). *Biochim. Biophys. Acta*, 1958, **27** (1), 184–188.—The method is based on the reaction of pentoses and higher sugars with strong H_2SO_4 to give breakdown products which have characteristic u.v. absorption spectra and which combine with cysteine to give coloured products; tetroses do not give the reaction, but can combine with the breakdown products of higher sugars to give coloured compounds with characteristic absorption spectra in the visible region. Amounts of 5 to 50 μg of tetrose are determined after reaction with fructose and H_2SO_4. Higher sugars give different colours after addition of cysteine; glycollaldehyde gives a slight colour, but trioses do not react. W. H. C. SHAW

3445. **Use of resorcinol for identification and determination of monosaccharide groups. A report on a Gaucher spleen cerebroside.** A. W. Devor, C. Conger and I. Gill (Ohio State Univ., Columbus, U.S.A.). *Arch. Biochem. Biophys.*, 1958, **73** (1), 20–28.—The two colorimetric methods described are based on the use of resorcinol, (*a*) with, and (*b*) without, pre-sulphonation, and are suitable for distinguishing between galactose and mannose and between xylose and arabinose; glucose is distinguished from other common hexoses. Method (*a*) is preferred for qual. and (*b*) for quant. use. *Procedures*—(*a*) Mix 2 ml of aq. carbohydrate soln. (50 μg) with 5 mg of resorcinol. Add rapidly, with stirring, 5·5 ml of H_2SO_4 (100 ml of conc. H_2SO_4 added to 10 ml of water and cooled). Allow to stand for 45 min. and then determine the absorption curve (400 to 560 mμ) or measure the extinction at the appropriate max. (*b*) Add 100 ml of conc. H_2SO_4 to 10 ml of 2·1% aq. resorcinol. Allow to stand for 45 to 90 min. Rapidly add, with mixing, 5·5 ml of the reagent to 2 ml of aq. carbohydrate soln. in a test-tube. Place the tube in boiling water and remove after a total heating time of 5·5 min. Allow to stand for 45 min., then examine as in (*a*).
W. H. C. SHAW

3446. **The determination of hydroxypyruvate and glycollaldehyde.** F. Dickens and D. H. Williamson (Courtauld Inst. of Biochem., Middlesex Hosp. Med. Sch., London). *Biochem. J.*, 1958, **68** (1), 84–88.—A colorimetric method is described. When a soln. of each product is heated with naphtharesorcinol in 23 *N* H_2SO_4 the intensity of the grass-green colour, measured at 660 mμ, is proportional to the amount of the substance present under defined conditions. When both substances are present together, the determination is repeated after adsorption of the hydroxypyruvic acid on Bio-Deminrolit G. Glyoxylic acid can be determined under the same conditions as it gives a well-defined max. at 480 mμ. Among other substances tested only dihydroxymaleic acid interferes. A method is also described for the determination of hydroxypyruvate and pyruvate singly or together by the use of hydroxypyruvate reductase from parsley leaves and lactic dehydrogenase from muscle, both in the presence of reduced diphosphopyridine nucleotide. The reductase reacts only with hydroxypyruvate, whereas the dehydrogenase reacts equally well with both substrates. The reactions can be followed photometrically. J. N. ASHLEY

3447. **The occurrence of *p*-hydroxyphenylpyruvic and other keto acids in the urine of healthy and cirrhotic rabbits.** R. Ammon and U. Henning (Physiol. Chem. Inst., Univ. Saarlandes in Homburg). *Hoppe-Seyl. Z.*, 1957, **306**, 214–220.—The examination of urine for *p*-hydroxyphenylpyruvic and other keto acids by paper-chromatographic separation of their 2:4-dinitrophenylhydrazones is complicated by the fact that *cis* - *trans* isomerism causes more than one spot to be given by each acid. More satisfactory results are obtained if the extract of the 2:4-dinitrophenylhydrazones in $CHCl_3$ - ethanol (4:1, by vol.) is evaporated to dryness and, after being dissolved in ethanol, is reduced by tin and HCl gas for 7 to 8 hr. This process gives amino acids corresponding to the original keto acids. The excess of Sn is pptd. with H_2S. The filtrate is dried *in vacuo* and, after being dissolved in a few ml of water, is applied to a column of Amberlite resin from which the amino acids are eluted with *N* aq. NH_3. After evaporation of the eluate, two-dimensional chromatography of the amino acids is carried out. By this technique the occurrence of *p*-hydroxyphenylpyruvic acid has been shown in healthy and cirrhotic rabbits. Phenylpyruvic, *p*-hydroxyphenylpyruvic, *p*-hydroxyphenyl-lactic and *p*-hydroxyphenylacetic acids have also been identified in healthy human urine. F. POWELL

3448. **Influence of phenothiazine and some of its derivatives on the determination of 5-hydroxyindolylacetic acid in urine.** G. Ross, I. B. Weinstein and B. Kabakow (Montefiore Hosp., New York, U.S.A.). *Clin. Chem.*, 1958, **4** (1), 66–76.—Administration of phenothiazine deriv. (*e.g.*, promazine, promethazine or chlorpromazine) interferes with the chemical determination of 5-hydroxyindolylacetic acid in urine. Other substances were subsequently found to have a similar effect. It is suggested that all medication should cease for four days before collection of the specimen to be analysed.
H. F. W. KIRKPATRICK

3449. **A study of the identification and determination of phenols and phenolic acids in urine.** S. L. Tompsett (Dept. of Clin. Chem., Univ. of Edinburgh, Scotland). *Clin. Chim. Acta*, 1958, **3** (2), 149–159.—The Folin - Ciocalteu, indophenol pigment formation, 1-nitroso-2-naphthol, and Ehrlich reactions are studied and their application to urine extracts after paper chromatography is described. The concn. of some phenolic substances in normal urine are tabulated. H. F. W. KIRKPATRICK

3450. **Use of charcoal to separate mixtures of inorganic, ester and nucleotide phosphates.** R. K. Crane (Washington Univ. Sch. of Med., St. Louis, Mo., U.S.A.). *Science*, 1958, **127**, 285–286.—Ester and nucleotide phosphates are adsorbed on acid-washed Norit-A from 0·05 *N* HCl. Ester phosphates may be recovered by extraction with 10% trichloroacetic acid soln., and nucleotide phosphates by extraction with a mixture of 25% trichloroacetic acid (4 parts), and ammonium trichloroacetate soln. (6 parts) prepared by adding 8·3 ml of 28% w/v aq. NH_3 to 20 ml of 100% w/v trichloroacetic acid.
H. F. W. KIRKPATRICK

3451. **Determination of organic compounds below the microgram range. Ribonucleic acid and its constituents.** J.-E. Edström (4 Langgatan 7, Göteborg C, Sweden). *Microchem. J.*, 1958, **2** (1), 71–82.—A review is presented of methods that have been adapted for the determination of ribonucleic

acid (**I**) and its nucleotides in single animal cells. The quantitative determination of **I** in the range 25 to 5000 $\mu\mu$g and of nucleotide by means of micro-electrophoresis down to 200 $\mu\mu$g may be undertaken. D. F. PHILLIPS

3452. **Quantitative determination of nucleic acids in whole tissue by paper chromatography.** W. J. Kleinschmidt and J. A. Manthey (Lilly Res. Lab., Indianapolis, Indiana, U.S.A.). *Arch. Biochem. Biophys.*, 1958, **73** (1), 52–63.—The method described for the determination of ribo- and deoxyribonucleic acids is based on the hydrolysis of 50 to 100-mg amounts of the dried defatted tissue sample. The base constituents of the nucleic acids are then separated by two-dimensional paper chromatography, which also serves to eliminate contaminating hydrolysis products. The appropriate areas of the developed chromatogram are cut out and eluted with *N* HCl, and the extinction is measured at the wavelength of max. absorption for the particular base. A statistical examination of the accuracy of replicate results is made and the significance of assays of ribonucleic acid is discussed in relation to its structure. W. H. C. SHAW

3453. **Recent work in the quantitative determination of pituitary gonadotrophins in urine.** J. A. Loraine [Clin. Endocrinol. Res. Unit (M.R.C.), Univ., Edinburgh]. *Acta Endocrinol.*, 1957, Suppl. **31**, 75–84.—Assay methods and the extraction of gonadotrophins from human non-pregnant urine are reviewed. The use of human menopausal gonadotrophin as a standard preparation for the assay of pituitary gonadotrophins is proposed. A method for the routine estimation of gonadotrophins in human non-pregnant urine is presented, and some clinical applications of gonadotrophin assays are described. B. P. BLOCK

3454. **Rapid quantitative estimation of gonadotrophins in non-pregnant urine.** L. A. M. Stolte, J. M. J. Bukker and J. C. Seelen (Univ. Dept. of Gynaecology and Obstetrics of Utrecht and Nymegin). *Acta Endocrinol.*, 1957, Suppl. **31**, 85–89.—Immature female mice are injected with a single dose of urine ultrafiltrate. The animals are killed after 18 hr. when the increase in uterine wt. is estimated on a quantal basis. B. P. BLOCK

3455. **Studies on fibrinolysin using a clinically practical method of quantitative determination.** E. Adelson and W. H. Roeder (Walter Reed Army Inst. of Res., Washington, D.C., U.S.A.). *J. Clin. Path.*, 1958, **11** (1), 82–86.—The method of Bidwell and Macfarlane (*Biochem. J.*, 1951, **49**, xiii; 1953, **55**, 497) is modified, with improved quant. application. H. F. W. KIRKPATRICK

3456. **Application of the Monche apparatus for micro-determination of blood gases to the determination of amino acids by their amino groups.** C. García del Amo (I. E. Fisiol. Bioquím., Madrid). *Rev. Esp. Fisiol.*, 1957, **13**, 35–46.—The modified Van Slyke apparatus described by Monche (*Ibid.*, 1953, **9**, 129) was further modified by introduction of an internal magnet of nialco. The determination of amino acids depends on the decomposition of amino groups and determination of amino nitrogen. Samples containing 0·01 mg of N can be used with an error $< 1\%$. NUTR. ABSTR.

3457. **Manometric evaluation of amino acids based on determination of their carboxyl groups.** C. García del Amo (I. E. Fisiol. Bioquím., Madrid). *Rev. Esp. Fisiol.*, 1957, **13**, 47–57.—With the modified Van Slyke apparatus (*cf. Anal. Abstr.*, 1958, **5**, 3456), amino acids are made to react with ninhydrin at 100°, and the CO_2 liberated is determined manometrically. Values for nitrogen and carboxyl carbon calculated from the data were compared with those found for amino nitrogen. By comparing the data for a mixture of two or more amino acids the proportion of each in the mixture can be deduced. NUTR. ABSTR.

3458. **Amino-acid determination on paper chromatograms.** J. Heilmann, J. Barrollier and E. Watzke (Hauptlab. der Schering A.-G., W. Berlin). *Hoppe-Seyl. Z.*, 1957, **309**, 219–220.—An improved Cd-containing ninhydrin reagent is described, which comprises 100 mg of Cd acetate, 10 ml of H_2O, 5 ml of acetic acid, 100 ml of acetone and 1 g of ninhydrin. Chromatograms are dipped in the reagent and colour development should take place in a closed chamber containing a dish of conc. H_2SO_4 to minimise background colour. The zones are eluted with methanol and the extinction is read at 500 mμ. The molar extinction coefficients of the individual amino acids vary widely, however, and difficulties are encountered with glycine, cystine and cysteine because of incomplete extraction. The separation of slow-running amino acids with similar R_F values may be considerably improved by repeated runs in the same solvent, but if the R_F values are originally above 0·5 this treatment is of no use. F. POWELL

3459. **Ion-exchange chromatography of amino acids. Effect of resin particle size on column performance.** P. B. Hamilton (Alfred I. du Pont Inst. of the Nemours Foundation, Wilmington, Del., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 914–919.—A back-washing technique is described for grading spherical or irregularly shaped particles of sulphonated polystyrene cationic resins, approximately 8% cross-linked, into 20 μ, 25 μ, 30 μ, etc., diameter classes, each having 60 to 75% of the particles within $\pm$ 3 μ of the average diameter of each class, and a diameter range of $\simeq \pm$ 7 μ. Separation of amino acids was optimum with spherical particles of 25 μ or irregular particles of 30 μ diam. A range of 20 to 40 μ was equally useful provided that size distribution was uniform throughout the range. G. P. COOK

3460. **The determination of hydroxyproline in liver.** I. G. Fels (Hektoen Inst. Med. Res., Cook County Hosp., Chicago, U.S.A.). *Clin. Chem.*, 1958, **4** (1), 62–65.—The method of Neuman and Logan (*Brit. Abstr. C*, 1950, 412) is modified by adsorption of tyrosine on Norit charcoal, allowing determination of hydroxyproline to be made without correction for tyrosine. H. F. W. KIRKPATRICK

3461. **Paper-chromatographic determination of phenylalanine in serum.** H. Zahn and H. Marstaller (Univ. Kinderklinik, Würzburg). *Hoppe-Seyl. Z.*, 1958, **310** (1–2), 44–49.—The determination of phenylalanine can be carried out on untreated cerebrospinal fluid or urine or on the protein-free filtrates obtained by treating 1 vol. of serum with 4 vol. of ethanol. The error is of the order of $\pm$ 10%, but the chromatographic method is more accurate than standard colorimetric procedures for

low concn. of phenylalanine. The ethanolic extract is concentrated in a specially constructed centrifuge tube with an elongated narrow portion at the bottom. The concentrate is applied to the paper over a surface of 19 mm × 17 mm and chromatography is carried out in phenol or *o*-cresol buffered to pH 6·2. The chromatogram is treated with ninhydrin and the coloured product is converted into the copper derivative. The strip corresponding to phenylalanine is eluted with methanol and the extinction is read at 490 mμ. F. POWELL

3462. **Thymidine determination by some microbiological assay methods.** E. M. Lansford, W. M. Harding and W. Shive (Texas Univ., Austin, U.S.A.). *Arch. Biochem. Biophys.*, 1958, **73** (1), 180–190.—A description is given of culture media and procedures for three methods based on the growth response to thymidine of (*a*) *Leuconostoc mesenteroides* 8293 in the presence of methylfolic acid, (*b*) *Lactobacillus arabinosus* 17–5 in the presence of sulphanilamide, and (*c*) *Lb. arabinosus* in the presence of 2:4-diamino-6:7-diphenylpteridine as inhibitors. In method (*c*) a suitable response is obtained at concn. <0·001 μg per ml to about 0·1 μg per ml; no response was obtained from the other compounds tested at comparable concn. The thymidine contents of extracts of various natural products assayed by the three methods are listed. W. H. C. SHAW

3463. **The colorimetric determination of dipeptides by means of oxygenated cobalt complexes.** E. M. Crook and B. R. Rabin (Dept. of Biochem., Univ. Coll., London). *Biochem. J.*, 1958, **68** (1), 177–182.—The method, which is rapid, accurate, and applicable to a large number of dipeptides, depends on the production of coloured Co complexes, which are formed from dipeptides in the presence of alkaline $Co_3(PO_4)_2$ and H_2O_2. The intensity of the coloration produced by heating the mixture in boiling water is read in a colorimeter with an Ilford 624 (green) filter. Macro and micro modifications are described and 30 estimations can be carried out in 2 to 3 hr. The method is useful for following the hydrolysis, by acids, bases or enzymes, of dipeptides of monoaminomonocarboxylic acids, but not for dipeptides that contain cysteine, tyrosine, tryptophan or histidine residues, since these amino acids give appreciable colours under the conditions of the assay. Most other amino acids and ammonium salts cause no appreciable interference. J. N. ASHLEY

3464. **Quantitative micro-determination of proteins and peptides.** H. Nielsen (Dr. H. Nielsen's Lab., Aabyhøj, Denmark). *Acta Chem. Scand.*, 1958, **12** (1), 38–43.—A protein - copper complex (biuret reaction) does not give a reproducible colour, but the copper in it can be determined colorimetrically as the diethyldithiocarbamate on the assumption that each atom of Cu corresponds to 4 atoms of protein N. *Procedure*—Plasma or serum (1 ml) diluted 50 times with normal saline, or cerebrospinal fluid (1 ml) undiluted, is treated with 2 ml of 10% aq. trichloroacetic acid soln. and the pptd. protein is centrifuged and dissolved in 5 ml of 5% aq. $Na_3PO_4.12H_2O$ soln. One drop of 4% aq. $CuSO_4.5H_2O$ soln. is added and, after shaking the mixture for 30 min., the excess of Cu as phosphate is centrifuged off and the soln. of the protein complex filtered through a sintered-glass disc. The filtrate (1 ml) is mixed with 5 ml of 5% aq. $Na_3PO_4.12H_2O$ soln. and treated with a few crystals of Na diethyldithiocarbamate. After 15 min. the extinction is measured at 440 mμ. The method is also applicable to polypeptides with at least three amino-acid units. E. J. H. BIRCH

3465. **Studies on the amide and C-terminal residues in proteins. I. The characterisation [and determination] of the C-terminal residue.** A. C. Chibnall and M. W. Rees (Dept. of Biochem., Univ. of Cambridge, England). *Biochem. J.*, 1958, **68** (1), 105–111.—Reduction of the pre-esterified carboxyl groups of C-terminal residues in proteins with $LiBH_4$, followed by acid hydrolysis, give amino alcohols or hydroxyamino acids which are estimated by standard methods. The method is not completely reliable for proteins of high mol. wt. such as β-lactoglobulin because of some reductive cleavage of peptide bonds which occurs to the extent of 1 to 2% of the total peptide bonds. With proteins of low mol. wt., such as insulin, this is not serious. The method may also be of use with low-mol.-wt. peptides.

II. [Determination of] the ammonia nitrogen and amide nitrogen of various native protein preparations. A. C. Chibnall, J. L. Mangan and M. W. Rees. *Ibid.*, 1958, **68** (1), 111–114.—In the determination of the amide-N content of native proteins, the free NH_3 content must also be determined. In a protein of high mol. wt. this may be equivalent to one or more amide-N groups per mol. A method for the determination of this ammoniacal N is described. It depends on the fact that the protein is pptd. from ethanolic HCl by diethyl ether while the ammonium salts remain in soln. After evaporation of the ether the NH_3 is determined with ninhydrin in the Conway apparatus.

IV. Separation and quantitative determination of β-amino alcohols. M. W. Rees. *Ibid.*, 1958, **68** (1), 118–122.—Mixtures of β-amino alcohols are separated chromatographically on a column of silica gel buffered to pH 4·85 with phosphate buffer. Elution with mixtures of ethanol - chloroform gives 96 to 100% return of the amino alcohols. A method is described for the determination of these alcohols based on their oxidation to formaldehyde by HIO_4. The formaldehyde is determined colorimetrically at 570 mμ by the use of a standard chromotropic acid method.

V. Estimation of asparagine and glutamine residues. A. C. Chibnall, C. Haselbach, J. L. Mangan and M. W. Rees. *Ibid.*, 1958, **68** (1), 122–128.—A method is described for the determination of the total number of asparagine and glutamine residues in proteins. It is based on modifications that aspartyl, glutamyl, C-terminal asparaginyl and C-terminal glutaminyl residues undergo when esterified and then reduced with $LiBH_4$. C-Terminal asparaginyl and glutaminyl residues give β-amino-γ-hydroxybutyric acid and γ-amino-δ-hydroxyvaleric acid, respectively, whereas when these residues are in the chain they remain intact and after acid hydrolysis give aspartic and glutamic acids, respectively. Aspartyl and glutamyl residues in the chain are esterified at the ω-carboxyl groups, and reduction and hydrolysis then give α-amino-γ-hydroxybutyric acid and α-amino-δ-hydroxyvaleric acid, whereas if they occupy C-terminal positions the same sequence of reactions will give the corresponding amino-diols. These diols and β-amino-γ-hydroxybutyric acid and γ-amino-δ-hydroxyvaleric acid are oxidised by HIO_4 with formation of formaldehyde, which is determined by the chromotropic acid method. The aspartic and glutamic acids (which are determined

with ninhydrin), as well as β-amino-γ-hydroxybutyric acid and γ-amino-δ-hydroxyvaleric acid, are separated on Dowex-50 columns by elution with aq. HCl. The two diols are determined after separation on silica gel. J. N. ASHLEY

3466. **Determination of the N-terminal residues in proteins with methoxycarbonyl chloride [methyl chloroformate].** A. C. Chibnall and P. F. Spahr (Dept. of Biochem., Univ. of Cambridge, England). *Biochem. J.*, 1958, **68** (1), 135–142.—Methyl chloroformate has no particular advantage over 1-fluoro-2:4-dinitrobenzene as a reagent for the determination of N-terminal groups in proteins. It affords methoxycarbonylamino acids which, on subsequent hydrolysis of the condensed protein in 8·6 *N* HCl at 37°, are readily separated from other hydrolysis products on a buffered column of Celite. Good resolution is obtained by using gradient elution, and the acids are determined by titration with standard alkali. J. N. ASHLEY

3467. **Electrophoresis with isolated proteins.** H. E. Schulze (Behringwerke, Marburg a.d. Lahn, Germany). *Clin. Chim. Acta*, 1958, **3** (1), 24–33.—The use of isolated proteins of defined properties in electrophoretic studies is illustrated. It is possible to determine the influence of the use of different methods on the mobility, the effect of small amounts of accompanying substances on the electrophoretic behaviour of the proteins, and the degree of mutual interaction of the proteins in the various methods. H. F. W. KIRKPATRICK

3468. **Reactions for characterisation of constituents of biological liquids after electrophoresis on agar.** J. Uriel (Inst. Pasteur, Paris, France). *Clin. Chim. Acta*, 1958, **3** (1), 17–23.—The advantages and disadvantages of this medium in comparison with other media (paper, starch, etc.) are discussed. H. F. W. KIRKPATRICK

3469. **Two-dimensional electrochromatography and triple staining of normal serum.** Z. Pučar, M. Keler and M. Petek (Inst. Ructer Bošković, Zagreb, Yugoslavia). *Hoppe-Seyl. Z.*, 1957, **309** (1–3), 43–51.—By means of the apparatus already described (*cf.* Pučar and Petek, *Hoppe-Seyl. Z.*, 1957, **308**, 241) human serum was subjected to two-dimensional electrochromatography in order to study the lipid and carbohydrate distribution. Two electrochromatograms were run with 0·01 ml of serum, in each case using the same method as previously described for the separation of proteins. One chromatogram was then stained with Sudan black to show the lipids and the other with potassium periodate and fuchsine to show the carbohydrates. Each chromatogram was photographed and, in addition, the position of the spots was recorded by means of tracing paper. Both chromatograms were then stained with bromophenol blue or Amido black to reveal the proteins. The carbohydrates were always associated with the protein fractions, but the lipids showed little correlation with the proteins. The lipid distribution was different from that found in one-dimensional electropherograms, and evidence is presented that a lipid component may be associated at the conclusion of the process with a different protein fraction from that to which it was originally bound. F. POWELL

3470. **Polarographic determination of electrophoretic fractions.** E. Balle-Helaers (Lab. d'Analyses Médicales, Brussels, Belgium). *Clin. Chim. Acta*, 1958, **3** (1), 51–56.—Application of the Brdička protein wave to the evaluation of the protein fractions after electrophoresis is described. Development into an accurate estimation for concn. between 5 and 150 μg per ml of supporting electrolyte is a possibility if calibration is made with pure fractions taken through the entire procedure. H. F. W. KIRKPATRICK

3471. **Comparative investigation of lipoproteins in human serum and ultracentrifugates by zone electrophoresis on paper and agar.** F. A. Pezold (Med. Klin. der Freien Univ., West Berlin, Germany). *Clin. Chim. Acta*, 1958, **3** (1), 40–44.—Protein fractions on the two media correspond but lipoprotein fractions differ considerably. A decrease in α-lipoproteins in paper electrophoresis is not necessarily a real one, and a more reliable indication is given by the agar-gel method. H. F. W. KIRKPATRICK

3472. **Quantitative application of sample dispersion in potassium bromide for infra-red analysis of steroids.** H. Rosenkrantz, P. Potvin and P. Skogstrom (Worcester Foundation for Exp. Biol., Shrewsbury, Mass., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 975–977.—In this study of the i.r. analysis of steroid mixtures, a rectangular die was used for the preparation of the potassium bromide pellets. Plots of density *vs.* concn. for androgenic, oestrogenic and corticosteroid hormones and plots of the change in ratio of characteristic bands *vs.* the percentage concn. of two-component mixtures gave approx. linear relationships. Known mixtures were determined to within 8% of the actual concn. Sample size, solvent transfer and the particular steroid affected the acceptability of the spectra. G. P. COOK

3473. **Micro-estimation of serum cholesterol and esters on finger-tip blood.** H. L. Rosenthal and L. Jud (Rochester Gen. Hosp., N.Y., U.S.A.). *J. Lab. Clin. Med.*, 1958, **51** (1), 143–151.—The method described is suitable for 0·05-ml samples and gives results that compare favourably with those obtained by macro-methods. After extraction with a mixture of acetone and ethanol, an aliquot is treated with digitonin soln. and the total cholesterol is determined immediately from the extinction of the cholesterol - iron complex at 560 mμ. The estimation is repeated after 4 to 24 hr. on the supernatant liquid obtained after the free cholesterol has been pptd. as the digitonide. W. H. C. SHAW

3474. **Chemical determination of oestrogens in urine.** E. Diczfalusy (Dept. of Women's Diseases, Kardinska Sjukhuset, Stockholm). *Acta Endocrinol.*, 1957, Suppl. **31**, 11–26.—Several problems related to the estimation of urinary oestrogens are reviewed. All existing methods are briefly surveyed with the conclusion that chemical methods are preferable. New methods are needed for the separate estimation of the 6 or 7 oestrogens so far detected in human urine. B. P. BLOCK

3475. **The application of colour reactions to the identification of some progesterone derivatives.** F. Hohensee and R. Huttenrauch (Lab. des VEB Jenapharm, Jena). *Hoppe-Seyl. Z.*, 1958, **310** (1–2), 19–22.—Five colour reactions are described for the identification of progesterone derivatives as spot tests on filter-paper. A table is given showing the

colours observed in both visible and u.v. light when these reactions are applied to progesterone and twelve of its derivatives. F. POWELL

3476. **Determination of neutral steroids in human blood.** J. Tamm, I. Beckmann and K. D. Voigt (2nd Med. Clin., Univ., Hamburg, Germany). *Acta Endocrinol.*, 1957, Suppl. **31**, 219–225.—Extract the serum or plasma with $CHCl_3$ to produce extract *A* (Silber and Porter, *J. Biol. Chem.*, 1954, **210**, 923). Determine free 17-hydroxycorticosteroids by the Porter - Silber reaction. Estimate 17-oxosteroids by a micro-modification of the Zimmermann reaction (Voigt *et al.*, *Dtsch. Arch. Klin. Med.*, 1955, **202**, 1) after performing a Girard separation (Girard and Sandalesco, *Helv. Chim. Acta*, 1936, **19**, 1095). Add a fivefold amount of ethanol to the aq. phase and heat to 40° to precipitate proteins. Evaporate the ethanol and shake the aq. phase twice with ether. Divide the aq. phase into 2 parts. Acidify one half to pH 1 with conc. H_2SO_4 and extract continuously with ether for 24 hr. Wash the ether 3 times with approx. 0·1 vol. of 0·1 *N* NaOH and 4 times with 0·2 vol. of H_2O, dry over Na_2SO_4 and evaporate. After applying Girard and micro-Zimmermann tests, perform the $HClO_4$ reaction (Pontius *et al.*, *Acta Endocrinol.*, 1955, **20**, 19) and Allen's reaction on 3 aliquots. Adjust the other half to pH 4·6 and incubate for 48 hr. at 37° with 62,000 units of glucuronidase per 100 ml of starting material. Submit the ether extract after glucuronidase hydrolysis to the same colour reactions as *A*. B. P. BLOCK

3477. **Electrophoretic and chromatographic separation of urinary conjugated 17-oxosteroids soluble in *n*-butanol.** G. Cavina and L. Tentori (Inst. of Health, Rome, Italy). *Clin. Chim. Acta*, 1958, **3** (2), 160–164 (in English).—The sulphate esters and glucuronides of steroids are separable by paper electrophoresis. Extraction of the conjugates and separation by this method and by paper chromatography, and their quant. determination are described. H. F. W. KIRKPATRICK

3478. **Paper-chromatographic determination of 17-oxosteroids in urine.** J. Barrollier and J. Heilmann (Hauptlab. der Schering A.-G., W. Berlin). *Hoppe-Seyl. Z.*, 1957, **309**, 221–225.—Ascending chromatograms are run on Whatman No. 1 paper that has been drawn through dimethylformamide and then blotted between filter-paper. *cyclo*Hexane almost saturated with dimethylformamide is used as the mobile phase. Because of the low R_F values, three successive runs in the same direction are used. The Zimmermann reaction is then carried out on the paper to locate the ketosteroid zones, which are cut out and extracted with benzene - ethanol - acetic acid (10:10:1, by vol.). The original ketosteroids are thereby regenerated. The Zimmermann reaction is repeated in a volumetric flask whose contents are diluted to volume with dichloromethane after the colour development is complete. The extinction of the lower layer is then read at 520 mμ. The method has been tested for androsterone, dehydro*epi*androsterone and 3α-hydroxyaetiocholan-17-one and found to give from 100 to 104% recovery. F. POWELL

3479. **Study of the Norymberski methods for determination of 17-oxogenic steroids (17-hydroxycorticosteroids) in urine.** C. Sobel, O. J. Golub, R. J. Henry, S. L. Jacobs and G. K. Basu (Bio-Science Res. Foundation, Los Angeles, Calif., U.S.A.). *J. Clin. Endocrin. & Metabolism*, 1958, **18** (2), 208–221.—The indirect (bismuthate) and direct (borohydride) methods and the Zimmermann reaction have been studied; the most suitable procedures are described in detail. Oxidation with bismuthate is allowed to proceed overnight to ensure complete reaction. In urines containing much glucose, preliminary extraction of free and conjugated steroids by the method of Edwards *et al.* [*Mem. Soc. Endocrin.*, 1953, (2), 53] is preferred. H. F. W. KIRKPATRICK

3480. **A method for the quantitative fractionation of urinary 17-oxosteroids with some observations on steroid excretion during administration of ACTH and in the adreno-genital syndrome.** R. V. Brooks (Dept. of Chem. Path., St. Thomas's Hosp. Med. Sch., London). *Biochem. J.*, 1958, **68** (1), 50–57.—An extract containing the urinary 17-oxosteroid conjugates is first hydrolysed with β-glucuronidase, then continuously extracted with diethyl ether at pH 0·8. The liberated 17-oxosteroids are purified by treatment with a Girard reagent and the ketonic fraction is treated with $KHCO_3$ in aq. methanol to hydrolyse any acetates formed in the Girard separation. The ketonic product is then separated into two fractions by chromatography on Al_2O_3. The first fraction eluted contains the 17-oxosteroids with no O at C_{11} and the second fraction contains 17-oxosteroids with a C_{11} O-function. These two fractions are resolved into their components by paper chromatography, and the final determination is made by the Zimmermann reaction on the residues obtained after eluting the individual steroids from the paper. J. N. ASHLEY

3481. **Studies of adrenal corticosteroids. I. Estimation of plasma corticosterone and cortisol.** R. S. Ely, E. R. Hughes and V. C. Kelly (Coll. of Med., Univ. of Utah, Salt Lake City, U.S.A.). *J. Clin. Endocrin. & Metabolism*, 1958, **18** (2), 190–207.—Improved specificity in the original procedure of Sweat (*Anal. Abstr.*, 1954, **1**, 1944; 1955, **2**, 1627) is obtained by substitution of ethyl acetate for chloroform for extraction, Florisil chromatography for pentane - ethanol partition, and a tungsten light source for the mercury arc in producing excitation light. H. F. W. KIRKPATRICK

3482. **Fluorescence and absorption spectra of some corticosteroids in sulphuric and phosphoric acids.** J. W. Goldzieher and P. K. Besch (Southwest Foundation for Research and Education, San Antonio, Tex., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 962–967.—Both the fluorescence and absorption spectra are affected by acid concn., time and the temp. of the reaction, but can be made the basis of quant. procedures. K. A. PROCTOR

3483. **A micro-dialysis procedure for extraction and isolation of corticosteroids from peripheral blood plasma [determination of corticosteroids].** H. Kalant (Dept. of Path. Chem., Univ. of Toronto, Canada). *Biochem. J.*, 1958, **69** (1), 99–103.—A micro-modification of the method of Axelrod and Zaffaroni (*Arch. Biochem. Biophys.*, 1954, **50**, 347) is used to determine the concn. of the larger corticosteroid fractions in as little as 1 ml of peripheral blood plasma. A special micro-dialysis extraction cell is described; after extraction, the corticosteroids are isolated by ascending chromatography

on paper, located by u.v. contact photography, eluted, and measured by the modified fluorimetric method. J. N. ASHLEY

3484. **Fluorimetric measurement of adrenocortical steroids in concentrated acid solution.** H. Kalant (Dept. of Path. Chem., Univ. of Toronto, Canada). *Biochem. J.*, 1958, **69** (1), 93–98.—Very simple and extremely sensitive methods are described for the determination of certain corticosteroids. They are based on the fluorogenic reaction of the steroids with very conc. H_3PO_4 or H_2SO_4. Methods are given for the routine determination of cortisol, corticosterone and cortisone, while modifications are suggested for the determination of aldosterone and 11-deoxycorticosterone. The fluorescence is measured in a Coleman Model 12 electronic photofluorimeter. J. N. ASHLEY

3485. **Quantitative determination of pregnane-3α:20α-diol in urine.** E. Verboom (Univ. Clin. Obstetr. Gynaecol., Utrecht). *Acta Endocrinol.*, 1957, Suppl. **29**, 1–50.—This absorptiometric method for the determination of pregnanediol in water and urine is based on the yellow - orange soln. produced when it is treated with conc. H_2SO_4. When dissolved in conc. H_2SO_4 many steroids yield yellow - brown - orange soln. The detailed investigation is described into the optimum conditions for the treatment of pregnanediol with conc. H_2SO_4 so that the resulting soln. possesses an absorptiometric spectrum whose ΔE reaches the max. possible. The influence of time, temp., acid concn., oxidising substances and sunlight was studied. *Procedure*—Adjust a 24-hr. urine sample to pH 2 with 25% HCl. Add Na pregnanediol glucuronidate (30 mg to 600 ml). Add 24 ml of HCl (25% w/w) and boil under reflux for 1 hr. in an oil bath at 130°. Cool the hydrolysate to room temp. and filter through paper. Dry the paper and extract with benzene - methanol soln. (1%) in a Soxhlet apparatus. Dry under vacuum and collect the residue in 50 ml of benzene containing 0·3% of methanol. Place 20 ml of this soln. on a column of Al_2O_3. Elute the column with 400 ml of benzene containing 0·3% methanol and 400 ml containing 0·6% methanol. Evaporate 5-ml samples to dryness and make up to 10 ml with conc. H_2SO_4. After heating in an oil bath at 100° cool the soln. and measure its extinction at 440, 420 and 380 mμ. The pregnanediol is calculated from a given formula. B. P. BLOCK

3486. **Identification of bile acids by paper chromatography.** O. Cerri and A. Spialtini (Ist. Sieroterap. Milanese "Serafino Belfanti," Milan, Italy). *Boll. Chim. Farm.*, 1957, **96** (5), 193–197.—Twenty bile acids were subjected to paper-chromatographic analysis; of these, 5 were conjugated and 15 were free bile acids or their esters. The analysis was carried out on Whatman No. 1 paper by ascending flow, with redistilled butanol freshly saturated with water and made alkaline with a few drops of conc. aq. NH_3. From 50 to 200 μg of material was used, giving spots of 0·5 cm in diam. on strips of paper 40 cm × 3 cm. The flow was stopped when the solvent had risen 25 cm, which took from 15 to 20 hr. according to the prevailing temp. The strips were air-dried and sprayed with a soln. of 0·9 g of vanillin dissolved in 15 ml of butanol containing 15 ml of conc. H_3PO_4, and finally dried in an oven at 70° for 10 min. Owing to the softening effect on the paper of the conc. H_3PO_4, the strips, after development, should be rapidly transferred to a basin of water. The R_F values and colours of the spots on development are given in tabular form; after numerous trials, the R_F values have been found never to vary by more than ± 0·01. It has been found that both a double bond (as in lithocholic or cholic acid) or spatial configuration (as in 3β-5-cholenic, dioxo- and dioxo*allo*-cholanic acids) may influence the position of the spots. P. HAAS

3487. **Measurement of enzymic amylolytic activity. Group B. I. Chemical methods.** H. Wildner and G. Wildner (Ireks Forschungsinst. f. Gärungswiss., Kulmbach, Germany). *Brauwissenschaft.*, 1958, **11** (1), 12–17.—Detailed descriptions are given of published methods for the following determinations—the diastase content of blood, the β-amylolytic activity of preparations of the enzyme, of grain (germinated and ungerminated) and of flour (by the polarimetric and "dextrin value" methods), and of β-amylase in soln. or in grain (with differentiation between the active and inactive states of the naturally-occurring enzyme). P. S. ARUP

3488. **Direct spectrophotometric measurement of the peptide bond: application to the determination of acylase I.** M. A. Mitz and R. J. Schlueter (Armour & Co., Chicago, Ill., U.S.A.). *Biochim. Biophys. Acta*, 1958, **27** (1), 168–172.—The simple rapid method described is based on the changes in the extinction values of peptides between 200 and 240 mμ that take place during enzymatic hydrolysis. From the rate of change in extinction at a suitable wavelength per min. the enzyme activity may be calculated by the method given. The assay of renal acylase I is detailed and the general applications of the method are discussed. W. H. C. SHAW

3489. **Determination of plasma amine oxidase activity.** F. L. Humoller, F. A. Majka, A. J. Barak, J. D. Stevens and J. M. Holthaus (V. A. Hosp., Omaha, Neb., U.S.A.). *Clin. Chem.*, 1958, **4** (1), 1–12.—A modification of the method of Akerfeldt (*Science*, 1957, **125**, 117) is described. H. F. W. KIRKPATRICK

3490. **A spectrophotometric method for the determination of bacterial hyaluronidase.** H. Greiling (Physiol. Chem. Inst., Freien Univ., Berlin). *Hoppe-Seyl. Z.*, 1957, **309**, 239–242.—The action of bacterial, but not of testicular, hyaluronidase causes the appearance of products that have a high absorption at 230 mμ. A method is described making use of this property which is specific for bacterial hyaluronidase and, in contrast to previously employed techniques, gives results that are independent of the degree of polymerisation of the hyaluronic acid. It is therefore suitable for use in kinetic investigations. F. POWELL

3491. **The analysis of hyaluronidase preparations.** M. Büchner and H.-C. Gabsch (1 Med. Klinik des Stadtkrankenhauses, Dresden-Friedrichstadt). *Pharm. Zentralh.*, 1958, **97** (1), 5–7.—Results are described which indicate the possibility of analysing hyaluronidase preparations and similar enzymes by means of paper electrophoresis and paper chromatography. The method used for paper electrophoresis was that usually applied to the analysis of proteins, and the optimum sample size was found to be a strip of about 4 cm × 22 cm. The fractions were developed with Amido black 10B for proteins, ninhydrin for free amino acids, and the periodic acid - Schiff reaction for glycoproteins. Tabulated

results on the commercial preparations "Kinetin" and "Hylase" indicate that it is possible to separate, and probably isolate, the active fractions by paper electrophoresis. An ascending paper-chromatographic method was applied to the same materials with the following solvents—butanol - glacial acetic acid - water (4:1:1) and (4:1:5); collidine; phenol - water (7:3); phenol - water in an atm. of NH_3. Results on the unhydrolysed materials showed that "Hylase" contains more free amino acids than "Kinetin," while the hydrolysed samples had substantially similar amino-acid compositions. The following amino acids were qualitatively identified —cysteine, R_F 0·09; arginine 0·14; taurine 0·15; glycine 0·18; aspartic acid 0·20; threonine 0·22; glutamic acid 0·26; alanine 0·28; proline 0·35; methionine 0·45; valine 0·48; phenylalanine 0·53; leucine - *iso*leucine 0·61. Polarographic examination of the samples also indicates typical concentration-dependent catalytic protein stages.

S. M. MARSH

See also Abstracts—**3378**, Detection of steroids. **3381**, Determination of lactaldehyde and related compounds. **3502**, Determination of oxytetracycline in blood. **3503**, Assay of insulin. **3530**, Paper chromatography of higher fatty acids. **3533**, Determination of riboflavine. **3534**, **3535**, Determination of vitamin B_{12}. **3540**, Analysis of radioactivity of biological samples. **3557**, A "micro" homogeniser for small-volume samples. **3572**, Anomalous i.r. spectra of steroids.

Drugs

3492. **Quantitative determination of codeine and amidopyrine by fractionation with "Kalignost."** M. Kranjčević and V. Broz-Kajganović (Inst. for Drug Control, Zagreb, Yugoslavia). *Croat. Chem. Acta*, 1958, **30** (1), 47–52.—Organic bases such as codeine (**I**) and amidopyrine (**II**) form complexes with "Kalignost" (Na tetraphenylboron), which on reaction with $HgCl_2$ liberate HCl; this reaction provides a method for the determination of these bases. It has been observed that the complex of **I** phosphate is pptd. from both weakly acid (pH 4 to 5) and weakly alkaline (pH 10) soln., but the complex of **II** is pptd. only from weakly alkaline soln. A method of separation and quant. analysis for **I** phosphate and **II** is described which is based on this difference in behaviour. Results are detailed for the analysis of the two compounds in a simple mixture, and also in pharmaceutical preparations containing in addition phenacetin and caffeine sodium benzoate. The method gives extremely accurate results and can be applied successfully on a micro scale.

S. M. MARSH

3493. **Micro-determination of tropane alkaloids.** M. Pöhm (Fabrik A. v. Waldheim, Vienna). *Mikrochim. Acta*, 1958, (1), 120–123.—Tropic acid and its derivatives in amounts < 1 mg can be determined colorimetrically by means of *p*-dimethylaminobenzaldehyde. The material is mixed with ether (120 ml) and 10% aq. NH_3 (1 ml) and set aside for 2 hr. The filtered extract (100 ml) is extracted with 0·05 *N* HCl. The HCl extract is made alkaline with NaOH and extracted with $CHCl_3$. This extract is evaporated to dryness, 3 drops of aq. Br are added and evaporated off, the residue is dissolved in methanol and again evaporated with 3 drops of aq. Br. After drying for 2 hr. over P_2O_5, the residue is treated with 7 drops of Wasicky reagent (a soln. of 1 g of *p*-dimethylaminobenzaldehyde in 9 g of 88% H_2SO_4) and set aside for 2 min. It is then heated for exactly 180 sec. in a boiling-water bath and cooled in ice for 15 sec. Acetic anhydride (5 ml) is added with stirring, and after 30 min. the extinction is measured at 500 mμ.

G. BURGER

3494. **Comparison of methods of determination of alkaloids in ergot.** W. Dębska and H. Speichert (Zakład Anal. i Technol. Leku Roślinnego, Poznań). *Biul. Inst. Roślin Leczniczych*, 1958, **4** (1), 17–25.—Methods for the determination of alkaloids in ergot given by the British, Polish, Russian, Czechoslovakian and Hungarian Pharmacopoeias are discussed. The British method gave the most accurate results. Of five micro-methods also quoted, Bekešy's method (*Biochem. Z.*, 1939, **302**, II, 187) was found to be simple and accurate. The results obtained enable the conditions necessary for satisfactory determinations to be defined.

H. DMOWSKA

3495. **Analysis of ergot alkaloids. II. Confirmation and separation of hydrogenated alkaloids.** H. Rochelmeyer, E. Stahl and A. Patani (Pharm. Inst., Johannes Gutenberg Univ., Mainz, Germany). *Arch. Pharm., Berlin*, 1958, **291** (1), 1–3.—When the dihydro-alkaloids are irradiated with u.v. light they are converted into compounds exhibiting yellow - green fluorescence and can be identified on a chromatogram. The fluorescence is intensified on a filter-paper impregnated with formamide.

M. H. SAWISTOWSKA

3496. **Paper-chromatographic evaluation of *Rauwolfia* species.** B. P. Korzun, A. F. St. André and P. R. Ulshafer (Res. Dept., Ciba Pharmaceutical Products Inc., Summit, N.J., U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1957, **46** (12), 720–723.—Wet the ground *Rauwolfia* root (3 g) with H_2O (2 ml), heat under reflux with benzene (12 ml) for 1 hr., filter hot and wash the filter with benzene (12 ml). Repeat the extraction. Evaporate the combined extracts under reduced pressure and dissolve the residue in $CHCl_3$ (5 ml). Develop 5-, 10- and 15-μl aliquots by descending chromatography on Whatman No. 1 paper with formamide - methanol (7:3) as stationary phase and benzene - saturated soln. of formamide in *cyclo*hexane (1:1) as mobile phase. After 5 hr., dry the chromatograms at 90° for 10 min. and compare the size and intensity of fluorescence of the spots under u.v. light with standards prepared from the pure alkaloids. This method has been used to determine the content of reserpine and the distribution of other alkaloids in 25 species of *Rauwolfia*. An extraction procedure for the isolation of reserpine is also described.

A. R. ROGERS

3497. **Polarographic determination of nicotine in cultivated tobaccos.** P. Nangniot and N. Melamed (Lab. de Chim. Anal., Inst. Agronom. de l'État, Gembloux, Belgium). *Chim. Anal.*, 1958, **40** (1), 3–7.—Cultivated tobaccos contain nicotine and nornicotine, both of which are reduced together polarographically, but nornicotine is converted into a nitroso derivative, non-volatile with steam, by HNO_2 (*cf.* Bowen and Bartel, *Analyst*, 1944, **69**, 155). Tobacco powder (10 g) is treated with 20 ml of alkali (33% aq. NaOH - 94% ethanol, 3:1), and shaken for 1 hr. with 50 ml of ether and 50 ml of

light petroleum (boiling range 40° to 60°). After being set aside for a further 30 min., 50 ml of the ether layer is extracted for 3 to 4 min. with 50 ml of 0·1 *N* HCl. For the determination of total alkaloid, 1 to 2 ml of the acid extract is mixed with either 50 ml of acetate buffer (pH 3·5), or 50 ml of Britton and Robinson's buffer (pH 10·6) plus 5 ml of 0·2% gelatin. The whole is made up to 100 ml and polarographed. The $E_{\frac{1}{2}}$ *vs.* the S.C.E. = −1·16 to −1·23, and −1·76 V, in the two buffers, respectively. Results are referred to a standard curve obtained with pure nicotine. For the determination of nicotine only, 5 ml of acid extract is neutralised to phenolphthalein with NaOH, and treated with 2 ml of 20% acetic acid and 0·5 g of powdered $NaNO_2$, and after 5 min. the mixture is made alkaline with NaOH and steam-distilled for 20 to 30 min. into a few ml of 0·2 *N* acetic acid or of Britton and Robinson acid mixture, according to the pH of polarography. After evaporation and making up to 50 ml, a suitable aliquot is polarographed as before. This gives nicotine only. The difference in wave-height between the two polarograms is referred to a standard curve obtained with nornicotine. Results for total alkaloids and for nicotine and nornicotine agreed well with those by the gravimetric tungstosilicic method of Bowen and Bartel.
R. E. Essery

3498. **The evaluation of the active principles of foxglove leaves by colorimetric methods.** Z. Kowalewski (Zakład Anal. i Technol. Leku Roślinnego, Poznań). *Biul. Inst. Roślin Leczniczych*, 1958, **4** (1), 1–17.—Four methods for the determination of cardiac glycosides in *Digitalis purpurea* and *Digitalis lanata* are discussed. The results are summarised and compared with those obtained by a biological method. Tattje's method was found to be the most satisfactory. H. Dmowska

3499. **Analysis of extracts of digitalis by paper chromatography.** L. Fauconnet and K. Kreis (Inst. Pharmacog., Univ. Lausanne, Switzerland). *Pharm. Acta Helv.*, 1957, **32** (6), 253–259.—The R_F values of a series of constituents of digitalis extracts have been determined by descending chromatography (on S. & S. paper No. 2045 A matt) with 2 solvent mixtures, namely, (*a*) dichloromethane - methanol - H_2O (11:4:5, by vol.) and (*b*) benzene - methanol - H_2O (2:2:1, by vol.), the lipophilic phase in each case being the mobile one. Dichloromethane was chosen in place of $CHCl_3$ because of its greater chemical stability, but, because of its volatility, the working temp. should be kept constant, within ±1°, especially during impregnation of the paper. The substances examined must be free from glycol or any non-volatile solvents. The spots are revealed by successive application of two soln. of trichloroacetic acid and the paper must be heated at 100° for 3 min. A 25% soln. of trichloroacetic acid in 95% ethanol reveals substances belonging to the B series which appear blue in u.v. light, and a 25% soln. of trichloroacetic acid in $CHCl_3$, to which a few drops of NaClO are added immediately before use, produces an orange - yellow fluorescence in the A series of compounds, and a white or green fluorescence in the B or C series of compounds, respectively. R_F values are tabulated. P. Haas

3500. **Determination of cantharidin in alcoholic tinctures.** E. Vevera (Pharm.-Chem. Inst., Tübingen Univ.). *Arch. Pharm., Berlin*, 1958, **291**; *Mitt. dtsch. Pharm. Ges.*, **28** (1), 4–7.—Shortcomings of assay methods for cantharidin, described in D.A.B. 6 and in Ph. Helv., are discussed and a quantitative paper-chromatographic method is described. (23 references.) M. H. Sawistowska

3501. **A cylinder-plate method for the quantitative determination of diffusion of antibacterial drugs from ointments.** E. M. Plein and J. B. Plein (Coll. of Pharm., Univ. of Washington, Seattle, U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1957, **46** (12), 716–720.—A reference soln. on each plate and a standard curve should always be used in the cylinder-plate method for the determination of antibacterial drugs in ointments. Straight lines are obtained by plotting the square of the diameter of the inhibition zone against the logarithm of the concn. of the following drugs—$HgCl_2$, chlortetracycline hydrochloride, oleandomycin phosphate, salicylic acid and sodium sulphacetamide.
A. R. Rogers

3502. **Colorimetric determination of tetracyclines by the use of a thorium reagent. II. Determination of oxytetracycline.** Takeichi Sakaguchi, Kiyomi Taguchi and Akira Suzuki (Pharm. Fac., Chiba Univ., Inohana-cho). *Japan Analyst*, 1957, **6** (12), 782–787.—The method for the determination of chlortetracycline with $Th(NO_3)_4$ (*cf.* Sakaguchi, *Anal. Abstr.*, 1956, **3**, 520) was applied to oxytetracycline (**I**). The max. absorption of the thorium chelate of **I** is at 385 mμ at pH 4·4, the extinction coeff. remaining unchanged at pH 3·6 to 5·4. The working curve is linear for < 25 μg of **I** per ml. **I** (20 to 250 μg) is extracted from urine (saturated with NaCl, containing 1 ml of 0·25 *M* EDTA, adjusted to pH 2 with 5 *N* HCl) with *n*-butanol (4 portions; 4, 2, 2 and 2 ml). The org. layer is washed with 25% NaCl soln. (1 ml) and shaken with 1% $Th(NO_3)_4$ soln. (a few portions; 2 ml each). The aq. layer is made up to 10 ml with 3 *M* acetate buffer (pH 4·0) (1 ml) and measured photometrically at 390 mμ. Extraction from blood is incomplete. Apo-oxytetracycline does not cause interference.

III. Determination of tetracycline. Takeichi Sakaguchi and Kiyomi Taguchi. *Ibid.*, 1957, **6** (12), 787–790.—A similar coloration occurs when $Th(NO_3)_4$ reacts with tetracycline (**II**), the max. absorption being at 395 mμ. The chelate is extractable into an aq. layer from soln. in *n*-butanol. **II** in urine is similarly extracted in the presence of EDTA and a large amount of NaCl. K. Saito

3503. **An assay of insulin by fibril formation from small samples of pancreas.** G. M. Grodsky (Dept. of Biochem., Univ. of Cambridge, England). *Biochem. J.*, 1958, **68** (1), 142–145.—The method, depending on the formation of fibrils, is applicable to 1-g samples of pancreas. Recoveries are linear and nearly quant. Although some modified insulins form fibrils, a general protein mixture from liver, as well as albumin and glucagon, does not interfere with the assay. The results obtained are comparable with those obtained by biological assay. No differences in insulin yield occur when seeding fibrils from ox, pig or sheep insulins are used. An apparatus is described for the simultaneous determination of 18 samples. J. N. Ashley

3504. **The assay of oxytocin in preparations of vasopressin.** A. T. Nielsen (Royal Danish School of Pharm., Copenhagen). *Dansk Tidsskr. Farm.*, 1958, **32** (1), 1–18.—This method depends on the

elimination of oxytocic activity due to the vasopressin itself, by the selective destruction of most of the vasopressin (<95%) by digestion with trypsin sufficiently dilute to exclude the possibility of oxytocic activity due to this enzyme. The chlorbutol which is used as a preservative is first removed by dilution of the sample (1 ml) with H_2O (1 ml) and evaporating gently to 1 ml. After the adjustment of the pH (with $NaHCO_3$) to 8·3, and the addition of trypsin (10 μg in a 0·1% soln.) the digestion is allowed to proceed at 22° for 30 min., and then stopped by bringing the pH to ≃3. The oxytocic activity of the resulting soln. is assayed by Holton's rat-uterus method. Amounts of oxytocin corresponding to 0·25 i.u. can thus be determined; values below this limit can be considered due to the oxytocic activity of the residual vasopressin. Reductions in oxytocic activity caused by the tryptic digestion of 9 samples of vasopressin correspond to an average activity of 3·8 oxytocic i.u. per 100 pressor units. P. S. ARUP

3505. **Use of tetra-*n*-butylammonium hydroxide for the analysis of barbituric acids.** D. E. Leavitt and J. Austian (Sch. of Pharm., Univ. Maryland, Baltimore). *Drug Standards*, 1958, **26** (1), 33–37.—Potentiometric titration of barbituric acid derivatives with tetra-*n*-butylammonium hydroxide in benzene gives a sharp inflection at the end-point when benzene - *iso*propyl alcohol (10: 1) or benzene - $CHCl_3$ is used as solvent. Visual titration in benzene - *iso*propyl alcohol with thymol blue as indicator gave high precision and recoveries of >99% with most barbiturates; recoveries of 98 to 99% were obtained with butobarbitone, methylphenobarbitone, allobarbitone and allylbarbituric acid. A. R. ROGERS

3506. **Determination of phemitone [methylphenobarbitone] in the presence of phenobarbitone by precipitation with sodium cobaltinitrite.** M. Kranjčević (Inst. for Drug Control, Zagreb, Yugoslavia). *Croat. Chem. Acta*, 1958, **30** (1), 53–61.—The use of sodium cobaltinitrite in the analysis of barbiturates is discussed with particular reference to methylphenobarbitone (**I**) and phenobarbitone (**II**). The pptn. of **I** from aq. soln. of Na **I** by sodium cobaltinitrite is due to a shift of the pH value from 9·3 to 9·4 to 5·4 to 5·6, and displacement of **I**, and not, as previously suggested, to complex formation. A method is described for the quant. determination of **I** in aq. soln. of Na **I** which is based on this pptn. reaction and which gives accurate and reproducible results. If sodium cobaltinitrite soln. is used to precipitate **I** from a mixture with **II**, some **II** is co-pptd. Two-stage recrystallisation of this mixture, in the first stage from the mother-liquors and in the second stage from water, both stages being carried out on a water bath for 45 to 60 min. at 70° to 80°, gave pure crystals of **I**. A method for the determination of **I** in its mixtures with **II** has been developed on the basis of these observations, and gives very good results. It is emphasised that freshly prepared soln. of sodium cobaltinitrite must always be used since with aged soln. errors might arise owing to the pptn. of $Co(OH)_2$. S. M. MARSH

3507. **Spectrophotometric titrations with nitrous acid. I. The estimation of procaine hydrochloride, propoxycaine hydrochloride, and tetracaine [amethocaine] hydrochloride.** E. L. Pratt (Control Lab., Winthrop Lab., Rensselaer, N.Y., U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1957, **46** (12), 724–728.—To determine procaine hydrochloride (**I**), propoxycaine hydrochloride (**II**) or tetracaine hydrochloride (**III**), dissolve the sample (400 mg of **I**, 1·5 mg of **II**, or 30 mg of **III**) in 6 *N* HCl (150 ml) and titrate spectrophotometrically at 385 mμ with 0·1 *N* $NaNO_2$. The end-point is marked by a change of slope in a graph of extinction (corrected for dilution if macro quantities are used) plotted against the vol. of titrant added. The change of slope indicates the presence of excess of HNO_2 in the titration of **I**, or the completion of formation of diazonium compound or nitroso derivative for **II** or **III**, respectively. The precision is better than 0·8% in all cases. The method has been applied to the determination of **I** and **III** in the presence of 3:4-dihydroxynorephedrine and a bisulphite compound. A. R. ROGERS

3508. **Spectrophotometric determination of cycloserine and isoniazid in pharmaceutical preparations.** J. M. Woodside, I. Piper and J. B. Leary (Anal. Lab., Eli Lilly and Co., Indianapolis, U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1957, **46** (12), 729–731.—Dilute the sample with 0·1 *N* HCl to contain ≃ 10 μg of cycloserine (**I**) per ml and measure the extinction at 219 mμ and 272 mμ with 1-cm cuvettes. The light absorption at 272 mμ is due to isoniazid (**II**) alone; that at 219 mμ is due to **I** and **II**. The method has an accuracy of ±2% and a precision of ±1%. Correlation with microbiological assay of **I** is good. A. R. ROGERS

3509. **A colorimetric procedure for iminazolines.** S. C. Slack and W. J. Mader (Res. Dept., Ciba Pharmaceutical Products Inc., Summit, N.J., U.S.A.). *J. Amer. Pharm. Ass., Sci. Ed.*, 1957, **46** (12), 742–744.—Laubie's colour test for iminazolines has been applied to the determination of 2-anilinomethyl-2-iminazoline hydrochloride (**I**), antazoline hydrochloride (**II**), tolazoline hydrochloride (**III**) and naphazoline hydrochloride (**IV**) in nasal soln. and tablets. The method is not applicable to phentolamine hydrochloride. The coeff. of variation is ±2%. *Procedure*—To the soln. (1 ml containing 0·25 mg of **I** or 0·5 mg of **II**, **III** or **IV**) add *N* NaOH (0·5 ml) and 5% sodium nitroprusside soln. (0·5 ml) and mix. After 10 min., add *M* $NaHCO_3$ (1 ml) and dilute with H_2O to 10 ml, and after a further 10 min. measure the extinction at 565 mμ against a reagent blank. A. R. ROGERS

3510. **Spectrophotometric determination in u.v. of cetyltrimethylammonium toluene-*p*-sulphonate in presence of interfering excipients.** B. Rocchi and F. Vittorelli (Lab. di Controllo, Carlo Erba S.p.A., Milan, Italy). *Boll. Chim. Farm.*, 1957, **96** (5), 198–201.—Following the spectrophotometric method described by Morton and Stubbs (*Analyst*, 1946, **71**, 348) involving the construction of a geometric correction curve, the authors have determined the amount of cetyltrimethylammonium toluene-*p*-sulphonate (**I**) in vaginal bougies containing excipients such as polyoxyethylene stearate or polyoxyethylene sorbitan stearate. The sample in a 1-litre flask is dissolved in 95% ethanol by gently warming on a water bath and the cooled soln. is made up to volume. Three spectrophotometric readings are then taken in 1-cm quartz cells at 217·5 mμ, 221·5 mμ and 224·2 mμ. From these readings it is possible to calculate the absorption

due to **I** and hence its wt. A series of determinations of 4 samples containing 25 mg of **I** gave values ranging from 23·5 to 26 mg with a mean of 24·925. The theory of the method is discussed.

P. HAAS

See also Abstracts—**3437**, Determination of morphine in blood and tissues. **3438**, Determination of barbiturates in body fluids. **3439**, Determination of sulphanilamide in blood serum. **3442**, Determination of adrenaline and noradrenaline in plasma. **3534**, **3535**, Determination of vitamin B_{12}. **3572**, Anomalous i.r. spectra of barbituric acids, steroids and alkaloids.

Food

3511. **Separation and estimation of sugars in cane products.** D. H. Foster and G. H. Marsh. *Int. Sugar J.*, 1958, **60**, 8–9.—A paper-chromatographic method is described by which sucrose, glucose and fructose from cane products can be quant. separated. The coeff. of variation for a single determination of sucrose was 1·12% when 13 mg was applied to the paper. No previous clarification of the soln. to be analysed was necessary for good separation. Unknown keto sugars in molasses prevented accurate determination of fructose content by selective oxidation methods. SUGAR IND. ABSTR.

3512. **Determination of ash by ion exchange. II. A rapid method of estimation of ash in clarified cane juice.** A. K. Chatterjee and S. Mukherjee. *Proc. 25th Ann. Conv. Sugar Technol. Assoc. India*, 1957, Part I, 87–89.—Samples of clarified cane juice of known Brix were passed through a column of strong cation-exchange resin (H form) and the resulting acidity was determined by titration after washing the resin with water. A general equation $Y = 0{\cdot}06552x + 1{\cdot}138$, where Y is the sulphated ash per 100 ml of 100° Bx. juice and x is the number of ml of normal acid released from the resin per 100 ml of 100° Bx. juice, was derived from a statistical analysis of the tabulated results. The differences between the calculated ash content and the ash content found by using this method ranged from −0·61 to +0·40 part of ash on a total of approx. 5 parts of ash per 100 ml of 100° Bx. juice; the runs can be completed in about 2 hr.

SUGAR IND. ABSTR.

3513. **Micro-determination of iron in cereals by direct extraction.** R. E. Salomon and E. M. Livingston (Brooklyn Coll., New York). *Microchem. J.*, 1958, **2** (1), 109–112.—The method proposed is designed to eliminate the initial lengthy step of dry-ashing, or, alternatively, of a digestion with conc. H_2SO_4 and HNO_3. Iron is reduced to the ferrous state by a 5% thioglycollic acid suspension. The soln. is then buffered with Na acetate - acetic acid and treated with 1:10-phenanthroline soln. to give a red coloration in the presence of Fe. The absorbance is measured at 510 mμ on a spectrophotometer. Samples of 1 g or less are used, and the Fe may be determined in its actual state of oxidation by omitting the thioglycollic acid suspension in one determination and adding it in another. The determination takes 15 min. There is no loss of ferric chloride or other volatile iron salts. The precision compares favourably with that attained by the ashing method.

D. F. PHILLIPS

3514. **Quantitative determination of peroxidase in sweet corn.** J. L. Vetter, M. P. Steinberg and A. I. Nelson (Dept. of Food Technol., Univ. of Illinois, Urbana, U.S.A.). *J. Agric. Food Chem.*, 1958, **6** (1), 39–41.—The sample is blended with phosphate - citrate buffer (pH 6·5) and incubated for 30 min. at 25°, then ethanolic *o*-phenylenediamine soln. and H_2O_2 are added, the enzyme activity being inhibited after 5 min. by adding saturated $NaHSO_3$ soln. The starch in the sample is flocculated by addition of ethanol and, after centrifuging, the extinction of the supernatant liquid is measured at 430 mμ. A reagent blank is prepared by adding the $NaHSO_3$ before the H_2O_2. The effect of blanching time on the peroxidase activity of sweet corn is recorded.

M. D. ANDERSON

3515. **Determination of micro quantities of methyl mercaptan [methanethiol] in γ-irradiated meat.** R. A. Sliwinski and D. M. Doty (American Meat Inst. Foundation and Dept. of Biochem., Univ. Chicago, Ill., U.S.A.). *J. Agric. Food Chem.*, 1958, **6** (1), 41–44.—Small amounts (5 to 110 μg) of methanethiol in γ-irradiated meat are determined by passing nitrogen for 4 hr. through a suspension of the ground meat in water at 58°, the vapours passing into a solution of 5% mercuric acetate at 0° to 4°. On addition of *p*-aminodimethylaniline and $FeCl_3$ in HNO_3 to this solution, methanethiol gives a red colour, which can be measured at 500 mμ. Hydrogen sulphide forms an insoluble complex with the mercuric acetate, and does not react with the amine to give methylene blue, so there is no interference. Methanethiol in ground raw beef increased with dosage of γ-radiation, up to 4 to 6·8 μg per g after a dose of 10×10^6 rep.

M. D. ANDERSON

3516. **Reviews of the progress of dairy science. Section C. Dairy chemistry. I. General and physical chemistry.** R. Aschaffenburg (Reading Univ., England). *J. Dairy Res.*, 1958, **25** (1), 125–132.—This review includes the physico-chemical properties of milk, and adulteration and its detection. (113 references.)

II. Milk composition and analysis and cheese ripening. E. R. Ling (Nottingham Univ. Agric. Sch., England). *Ibid.*, 1958, **25** (1), 132–142.—(131 references.) W. H. C. SHAW

3517. **Preservation of milk samples for analysis by different antiseptics.** M. Bejambes, G. Mocquot and P. Pauthe (Stat. Centr. Microbiol. Recherches Laitières, Jouy-en-Josas, France). *Lait*, 1957, **37**, 484–501.—In milk preserved by adding $K_2Cr_2O_7$, the lactose content decreased by approx. 10% and that of non-protein nitrogen (**I**) increased by 68% in 7 days and the milk coagulated. With phenylmercury borate as preservative the amount of **I** trebled in 8 days, but the lactose was unaltered. With a combination of formaldehyde and paraformaldehyde, lactose remained constant, but **I** increased by from 13 to 74% after 15 to 61 days, respectively. The last was the best preservative, but none was completely satisfactory.

NUTR. ABSTR.

3518. **A rejection test for raw milk.** P. B. Taylor and L. F. L. Clegg (Nat. Inst. Dairy Res., Reading Univ., England). *J. Dairy Res.*, 1958, **25** (1), 32–50.—The method is based on the pptn. of milk proteins with $BaCl_2$, NaOH and $ZnSO_4$, and the determination of apparent lactic acid in the filtrate by means of the yellow colour developed with $FeCl_3$.

The colour is measured against standards or permanent glass matching discs in a Lovibond comparator. Good relationship is attained with the keeping quality of milk (measured in hours to the clot-on-boiling end-point at 22°), and the value of 0·03% of apparent lactic acid is recommended as the lowest at which milk could be rejected for creamery purposes. W. H. C. SHAW

3519. **Influence of ration and time of feeding on the freezing-point and composition of cow's milk.** D. H. Kleyn, R. G. Warner, W. F. Shipe, W. K. Jordan, A. C. Dahlberg and R. F. Davis (Cornell Univ., Ithaca, N.Y., U.S.A.). *J. Dairy Sci.*, 1957, **40** (10), 1228–1237.—It is shown that the amount of roughage (hay) up to 100% in cow's rations has no effect on the daily average freezing-point of the milk, but that the feeding of roughage during the midday hours leads to greater water-consumption and to a freezing-point of the evening milk lower than that of the morning milk. Related variations in Cl^-, lactose, fat and solids-not-fat could not be detected. W. H. C. SHAW

3520. **Determination of unoxidised tocopherols in milk fat.** R. L. Handwerk and E. W. Bird (Iowa State Coll., Ames, U.S.A.). *J. Dairy Sci.*, 1958, **41** (1), 28–33.—The sample is saponified with methanolic KOH in the presence of pyrogallol to destroy tocoquinones and essentially all-fat peroxides. After extraction of the unsaponifiable matter with ether, the extract is dissolved in benzene and an aliquot is chromatographed on Floridin treated with $SnCl_2$ and HCl; this serves to remove carotenoids, any residual peroxides, steroids and vitamin A. Tocopherol is determined from the colour developed in a mixture of benzene and ethanol on treatment with $FeCl_3$ and dipyridyl in acetic acid. The method is suitable for following the oxidation of tocopherol in milk fat. W. H. C. SHAW

3521. **Determination of alginates, carboxymethylcelluloses and other thickening agents in milk derivatives and mayonnaise.** A. Fouassin (Lab. Recherches Bromatol., Univ. Liége, Belgium). *Rev. Ferment.*, 1957, **12**, 169–172.—The sample is treated with the Luff-Schoorl reagent ($CuSO_4$, Na_2CO_3 and citric acid), and casein is pptd. by adding acetic acid. Alginates, carboxymethylcellulose and other substances, such as Irish moss, gums, pectin and starch, present in the filtrate are pptd. by adding 1 vol. of conc. HCl and 2 vol. of ethanol and washed successively with ethanol and ether. A green colour is produced after boiling with anthrone and H_2SO_4 for 5 min. A method of separating alginates and carboxymethylcellulose is described. NUTR. ABSTR.

3522. **Identification and quantitative determination of diphenyl and thiourea in orange peel.** F. Hoeke and H. Cats (Keuringsdienst van Waren, Rotterdam, Netherlands). *Chem. Weekbl.*, 1957, **53** (46), 609–611.—*Procedure for diphenyl*—Inaccurate results with Steyn and Rosselet's determination (*cf. Analyst*, 1949, **74**, 89) are due to the ethereal oil present. It is found that Bruce and Howard's method for blood (*cf. Anal. Abstr.*, 1957, **4**, 1924) is applicable without interference from thiourea or Sudan II. Extract the orange in a covered container with $CHCl_3$ for 16 hr. then shake the extract with an equal vol. of 4 *N* HCl (× 2). Distil the $CHCl_3$ soln. with 1 ml of glacial acetic acid until a residue of about 2 ml is obtained, then warm this with 1 ml of conc. HNO_3 on a water bath (70°) for 1 hr. Add 10 ml of water (0°) and 1 ml of $CHCl_3$, shake for 2 min., remove the aq. layer with a pipette and wash twice with 10 ml of water. Transfer the $CHCl_3$ soln. to a 50-ml flask with 1 ml of 96% ethanol and 2·5 ml of 4 *N* HCl; warm for 3 min., then add 200 mg of zinc dust and warm for 10 min. on a boiling-water bath. Filter, and add successively 1 ml of 0·25% $NaNO_2$ soln., 1 ml of 2·5% $(NH_4)_2SO_4$ soln. and 2 ml of aq. 1% N-1-naphthylethylenediamine dihydrochloride soln., shaking and setting the mixture aside for 10 min. after each addition, then dilute to 50 ml. Measure the extinction at 570 mμ after 1 hr. against a blank. Prepare a fresh standard every time from 100 μg of diphenyl in 2 ml of $CHCl_3$. *Procedure for thiourea*—It was found that the colour reaction and extinction curve of the Grote method for the determination of thiourea (*cf. J. Biol. Chem.*, 1931, **93**, 25) are much the same for thiourea derivatives. To confirm their absence, an orange is leached with water for 16 hr. The extract is evaporated to 5 ml, coloured with 0·1 *N* iodine and cooled for 15 min. A 0·25% Na_2SO_3 soln. is added until the colour is discharged and the soln. is made up to 10 ml. A 5-ml aliquot is used for quant. determination of thiourea ("Methods of Analysis," A.O.A.C., 1955, 510); the rest is evaporated to dryness and dissolved in 2 drops of water. A paper chromatogram is made in the usual way with water-saturated $CHCl_3$ (S. & S., No. 2043b mgl). The R_F for thiourea is 0·0, and 1 for its derivatives. P. RENTENAAR

3523. **Analytical data on some common fruits.** R. W. Money *et al.* (Lyons Laboratories, 149 Hammersmith Road, London). *J. Sci. Food Agric.*, 1958, **9** (1), 18–20.—Data previously published (*J. Sci. Food Agric.*, 1950, **1**, 8) are revised and augmented. Figures for total solids, sugars, non-sugar solids, acidity, solids by refractometry, insoluble solids, pectin, pH, ratio of edible portion to skin, and average weight are tabulated. H. B. HEATH

3524. **Alkalimetric determination of free, combined and total sulphurous acid by means of the Lieb-Zacherl apparatus.** F. Paul (Höheren Bundeslehr- und Versuchsanstalten für Wein- und Obstbau, Klosterneuburg, Austria). *Mitt. Wein- u. Obstbau, Wien, A*, 1958, **8** (1), 21–27.—Minor modifications in the original design of the apparatus and analytical procedure (*Anal. Abstr.*, 1954, **1**, 3118) are described. P. S. ARUP

3525. **Determination of food tannins by ultraviolet spectrophotometry.** J. L. Owades, G. Rubin and M. W. Brenner (Schwarz Laboratories, Inc., Mount Vernon, N.Y.). *J. Agric. Food Chem.*, 1958, **6** (1), 44–46.—In this method, developed for determinations on beer and tea, and probably applicable to other materials, tannins are extracted from an acidified sample with ethyl acetate, the solvent is evaporated, the residue is heated at 80° to drive off any traces of solvent or of acetic acid, the tannins are taken up in acidified methanol, and the u.v. absorbance is measured at 270 mμ for beer and 275 mμ for tea. With beer, a preliminary extraction with *iso*octane to remove *iso*humulones is necessary before the extraction with ethyl acetate. M. D. ANDERSON

3526. **Methods of analysis of oils and fats.** British Standards Institution (2 Park St., London). B.S. 684: 1958, 100 pp.—Several additional methods of analysis are given in this revision of the 1950 edition of B.S. 684. These include the determination of melting-point, flow- and drop-points, cloud- and pour-points, smoke- and flash-points, titre, drying time, viscosity, Hanus iodine value, copper, polybromide value, and tests for various individual oils, namely for cottonseed (Halphen), sesame (Baudouin), and arachis (Evers). O. M. WHITTON

3527. **Fat rancidity. Summaries of papers presented at the second Scandinavian symposium on fat rancidity held in Elsinore, 9-11 Sept., 1957.** *Acta Polytech. Scand., Series Ch.* 1, 1958, (242), 72 pp.—The following summaries of papers are included. **Rapid determination of fat stability with regard to oxidative rancidity,** R. Marcuse, 24–29. **Stability tests in lard,** N. V. Jørgensen, 30–31. **Methods for the determination of peroxide values,** J. Glavind, 34–35. **Determination of peroxide in rye and rye crisp-bread,** G. Pein, 36–39. **Analytical control of oxidation during production and refining of oils and fats,** G. Wode, 51. N. E.

3528. **Determination of fat in meal.** J. Beneš (Res. Inst. of Food Technol., Prague). *Průmysl Potravin*, 1958, **9** (1), 56.—*Procedure*—Heat the ground sample (10 g) with H_2SO_4 (1:1) (20 ml) in a 250-ml flask till the meal dissolves. Cool, add water (20 ml) and trichloroethylene or CCl_4 (10 ml). Boil (5 min.) under reflux, transfer to a separating funnel, allow the layers to separate and filter the fat-containing portion through a filter-paper. Measure 25 ml of the filtrate into a weighed dish, evaporate the solvent on a water bath, dry the residue at 100° (1 hr.) and weigh. J. ZÝKA

3529. **Direct determination of saturated fatty acids in fats, oils and methyl esters.** D. F. Kuemmel (Procter & Gamble Co., Cincinnati, Ohio, U.S.A.). *J. Amer. Oil Chem. Soc.*, 1958, **35** (1), 41–45.—The fat (0·5 g) is heated under reflux with 10 ml of dry alkaline methanol for 1·5 hr. and the soln. is then neutralised to methyl orange with *N* HCl. The resulting methyl esters are extracted with $CHCl_3$ and the combined extracts are washed with 40 ml of water, 40 ml of 4% Na_2CO_3 soln. and a further 40 ml of water; the extract is dried over Na_2SO_4, then filtered, and the $CHCl_3$ is removed by evaporation under N. The methyl esters (0·5 g) are refluxed with 15 ml of acetone and the unsatd. esters are oxidised by careful addition of $KMnO_4$, after which $NaHSO_3$ soln. is added, followed by H_2SO_4 (1:4) until the soln. is blue to Congo red indicator. The satd. esters are then isolated by extraction with $CHCl_3$, and the solvent is finally removed, cooled and weighed. G. R. WHALLEY

3530. **Two-dimensional paper chromatography of higher fatty acids.** C. Michalec (Charles' Univ., Prague). *Biochim. Biophys. Acta*, 1958, **28** (1), 212–213.—Chromatography is carried out on Whatman No. 3 paper (15 cm × 15 cm) impregnated with a 5% soln. of liquid paraffin in diethyl ether. Development is by the ascending technique for 5 hr. at 20° with 93% aq. acetic acid. After drying the chromatogram at 80° to 90°, development in the second dimension is for 16 to 20 hr. at −8°, with a mixture of 85% formic acid - acetic acid - water (10:10:1, by vol.). The isolation of the higher fatty acids from blood serum is described. W. H. C. SHAW

3531. **Estimation of *trans*-isooleic acids in hydrogenated fats.** D. D. Nanavati, Sharda Dasgupta and J. S. Aggarwal (Nat. Chem. Lab., Poona, India). *J. Indian Chem. Soc.*, 1957, **34** (12), 885–888.—The accuracy of the Twitchell lead salt - ethanol method, as modified by Cocks *et al.* (*Analyst*, 1931, **56**, 368) is tested against the i.r. spectrophotometric method for the determination of *trans*-isooleic acids in fatty acid mixtures. In over 30 readings, in which the acid contents vary from 25 to 40%, the difference between the two methods varies from 0·1 to 6% of the infra-red reading. This warrants the use of the method when a spectrophotometer is not available. P. M. SORGO

3532. **Colorimetric determination of vitamin A with various chlorohydrins.** S. Erbe (Pharm. Inst., Martin Luther Univ., Halle-Wittenberg, Germany). *Z. anal. Chem.*, 1958, **159** (5), 327–332.—The suitability of 1-chloropropan-2-ol (**I**), 3-chloropropan-1-ol (**II**) and glycerol α-monochlorohydrin (**III**) as reagents for the colorimetric determination of vitamin A, in the ionising adsorption technique with $SbCl_3$, was investigated. Each reagent was activated by distillation *in vacuo* (10 to 20 mm Hg) after addition of 2% $SbCl_3$ as described by Sobel and Werbin (*Anal. Chem.*, 1947, **19**, 107). With **I** and **II**, maximum intensity of the weak green colour was achieved after heating for 2 min. at 60° to 70°. The colour with **I** was stable for 100 min. and with **II** for 60 min. With **III**, in the cold, a cornflower-blue colour that changed rapidly to violet was stable for from 5 to 15 min., but only for from 2 to 5 min. when warmed. **I** and **II** are considered to be better reagents than **III** because of their greater colour stability, although **III** develops a more intense colour. Maximum intensity for **I** and **II** is at 630 mμ and for **III** at 550 mμ. Correction factors are applied to compensate for deviation from the Beer - Lambert law, depending on the slope of the respective calibration curves. For **I**, μg of vitamin A acetate = $1585 \times \log I_0/I$ and for **II** = $2270 \times \log I_0/I$. Results differ from added amounts of vitamin A for **I** by ± 4%, **II** by ± 4·8% and **III** by ± 7·5%. Investigations into specificity and interferences are in progress. D. F. PHILLIPS

3533. **Determination of vitamin B_2 [riboflavine] in the presence of vitamin B_{12} and haematoporphyrin.** R. Ruggieri (Ist. Chim. Farm. Militare, Firenze, Italy). *Boll. Chim. Farm.*, 1957, **96** (6), 244–247.—A method is described for the colorimetric determination of the sodium salt of riboflavine phosphate in the presence of vitamin B_{12} and haematoporphyrin depending on the oxidation of the two last with $KMnO_4$ and destruction of the excess with H_2O_2. P. HAAS

3534. **Modified procedure for estimation of vitamin B_{12} in natural materials of low potency.** J. M. McLaughlan, C. G. Rogers, E. J. Middleton and J. A. Campbell (Food and Drug Lab., Dept. of Nat. Health and Welfare, Ottawa, Canada). *Canad. J. Biochem. Physiol.*, 1958, **36**, 195–201.—The method of Bacher *et al.*, for the spectrophotometric determination of vitamin B_{12} after extraction of

B_{12}-like factors, is modified for application to low-potency materials by using a shorter purification process, with extraction by metabisulphite instead of cyanide, and measuring vitamin B_{12} by means of *Lactobacillus leichmannii* **313**; the use of a radioactive tracer to determine loss during purification is replaced by a correction factor, shown by statistical analysis to be valid and reproducible. Purification removed pseudo-vitamin B_{12}, factor A and deoxyribosides that interfere with the assay by *Lb. leichmannii*, but vitamin B_{12} III was only partly removed. Purification did not affect determinations of vitamin-B_{12} activity in milk, blood plasma, chick mash, or fish meal. True vitamin B_{12} was apparently responsible for 50% of the total-B_{12} activity of dried cattle faeces, and for less than 10% of that of yeast extract. The coefficient of variation of the modified method was 8·7% based on 20 potency estimates. M. D. ANDERSON

3535. **Determination of vitamin B_{12} with *Escherichia coli* mutant.** J. Aronovitch and N. Grossowicz (Hebrew Univ., Jerusalem, Israel). *Clin. Chem.*, 1958, **4** (1), 22–26.—The method described employs a more sensitive mutant of *E. coli* that enables 4 to 20 $\mu\mu$g of vitamin B_{12} per ml to be determined. Comparison of the results of assays with this organism of human sera and animal tissue extracts with those given by the more specific *Ochromonas malhamensis* indicates no significant difference. The more rapid and simple method with *E. coli* is recommended for clinical use.

H. F. W. KIRKPATRICK

See also Abstracts—**3433**, Determination of ^{90}Sr and ^{140}Ba in food. **3487**, Measurement of amylolytic activity. **3548**, Determination of water in yeast cells. **3553**, Determination of "crude fibre."

Sanitation

3536. **Rapid semi-micro methods for chemical and industrial hygiene control of technical operations.** T. Iwanoff. *Chem. Tech., Berlin*, 1958, **10** (1), 35–40.—Methods for the determination of small amounts of poisonous vapours likely to occur in industrial operations are described. Suitable rapid methods for studying aniline vapour, nitrous and nitric oxides, hydrogen cyanide, benzene and mineral oil vapours, and carbon monoxide in air are discussed and particular methods are recommended. (21 references.) C. A. FINCH

3537. **Determination of calcium in natural waters.** J. H. Carpenter. *Limnol. & Oceanogr.*, 1957, **2**, 271–280.—A rapid and accurate procedure for the determination of Ca in natural waters involves photometric titration with EDTA (disodium salt) in the presence of murexide, after isolation of the Ca by ion-exchange chromatography. Calcium is separated from Mg and Sr by using acetylacetone as a complexing eluent. Use of the method to determine Ca in sea water showed that the calcium concn. was greater than would be expected from the Ca to Cl^- ratio for sea water. Previously the calcium concn. has been determined by the triple oxalate method, in which it is necessary to make a correction for Sr, and recent work has shown that the previously accepted value for Sr was too high, and therefore the corrected calcium concn. was too low. WATER POLLUT. ABSTR.

3538. **Colorimetric determination of zinc in water using Brilliant green and thiocyanate.** G. U. Houghton. *Proc. Soc. Water Treatm. Exam.*, 1957, **6**, 60–65.—A sensitive method has been developed for the determination of zinc in drinking water, based on the reaction with Brilliant green in acid soln. containing thiocyanate. Details are given. The method has an accuracy of ±8%. No interference is caused by up to 10 p.p.m. of Al and Pb, and little interference is caused by the presence of up to 3 p.p.m. of Cu and Fe.

WATER POLLUT. ABSTR.

3539. **Determination of fluorine in waters containing excessive amounts of interfering ions.** P. Venkateswarlu and D. Narayanarao (Dept. Biochem., Med. Coll., Trivandrum). *Indian J. Med. Res.*, 1957, **45**, 273–275.—Water from ponds and tanks (200 ml) was made just alkaline and boiled with 0·2 g of MgO for 10 min. The fluoride adsorbed on the MgO was distilled and titrated by conventional methods. The results were similar to those obtained by evaporation of the water before distilling; results by direct titration (*cf.* Venkateswarlu *et al.*, *Ibid.*, 1953, **41**, 253) were far too high owing to the presence of phosphate.

NUTR. ABSTR.

3540. **Analysis of radioactivity in surface waters [and biological samples]—practical laboratory methods.** L. R. Setter, G. R. Hagee and C. P. Straub (Taft Sanitary Engng Centre, Cincinnati, Ohio, U.S.A.). *Bull. A.S.T.M.*, 1958, (227), 35–40.—In the procedures described, the samples are prepared for counting by immediate filtration, evaporation of filtrates, drying at 103° (dry solids) or ignition at 600° (fixed solids) and, for most soils and biological samples, deposition of a thin film of the fixed solids on aluminium dishes (2-in. diam.). Milk and tissues are best processed by wet-ashing with conc. HNO_3. The counts of total α- and β-activity are made in an internal, gas-flow (CH_4 - A) proportional counter having a max. efficiency of $\simeq$50% for α-activity and 65% for β-activity from mixed fission-products one year old. The methods are valid for non-volatile radioactivity at levels above and well below 100 mC per litre (max. permissible limit in drinking-water in U.S.A.). The health significance of the observed activity can be interpreted from decay measurements or known radio-nuclide composition. W. J. BAKER

3541. **Determination of cyanides in industrial waste waters.** K. Zdeněk. *Korose a Ochrana Materiálu*, 1957, **1** (2), 26–28.—The method is applicable to the determination of cyanides in concn. < 3 mg per litre. Sulphides in concn. > 1 mg per litre interfere and must be eliminated by pptn. with Pb acetate. The determination is based on the reduction in alkaline soln. of picric acid by cyanides to give a red complex, which can be measured by using a 520-mμ filter or an appropriate dilution of 0·1 *N* $K_2Cr_2O_7$. For standards, aliquots of a soln. containing 0·04 mg of CN^- per ml are diluted to 15 ml with H_2O, 5 ml of 0·5 *M* Na_2CO_3 and 5 ml of 1% picric acid are added and the mixture is boiled for 5 min., then diluted to 100 ml with cold H_2O, and a curve is plotted. For low concn. of CN^-, a distillation method was developed. The cyanide-containing soln. is acidified with an excess of 2 ml of 2 *M* H_2SO_4 and the released HCN is absorbed in 0·5 *M* Na_2CO_3 and 1% picric acid in a separate vessel. CHEM. ABSTR.

3542. **The monitoring of effluent for alpha-emitters. III. Radium.** E. N. Jenkins and G. W. Sneddon (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 2385, 1958, 19 pp.—The Ra in the sample (150 ml) is co-pptd. with $PbSO_4$ (≡ 100 mg of Pb) which is redissolved in HCl - diethyl ether. By adding $Ba(NO_3)_2$ (≡ 5 mg of Ba), the Ra is co-pptd. with $BaCl_2$ which is dissolved and mounted on a glass counting-disc and then converted into $BaSO_4$, which is α-counted in a scintillation counter. Decontamination factors for U, Th, Pu and Po are >50. The limit of detection is ≃ 1 μμC per litre. Radium-226 may be determined from α-activity growth measurements on the source.

G. J. HUNTER

3543. **A method for the determination of radio-calcium and radio-strontium in effluent.** B. A. Loveridge (A.E.R.E., Harwell, England). A.E.R.E. Report C/R 1902, 1958, 21 pp.—The content of Ca in an aliquot of effluent is determined by an EDTA titration; if the phosphate concn. is > 10 p.p.m. the effluent is first passed through a column of De-Acidite-E resin (Cl form). Calcium is then added to another aliquot (400 to 600 ml) to bring the content of Ca to between 60 and 75 mg (as $CaCO_3$). Calcium phosphate is pptd. and re-dissolved; any residue is treated with HNO_3 and $HClO_4$, the soln. is added to the main soln., the solid is mounted and β-counted. The Ca is then re-pptd. as $Ca_3(PO_4)_2$ and redissolved; it is recovered by adsorption on a column of Zeo-Karb-225 resin and elution with 5 *N* HCl. After scavenges with Sb_2S_3, CoS and $Fe(OH)_3$, $CaCO_3$ is pptd. The ppt. is β-counted and all the activity is regarded as being due to ^{45}Ca. It is recommended that a flow counter be used since any over-estimation of Sr activity is thereby minimised. G. J. HUNTER

See also Abstract—**3320**, Determination of persulphate, H_2O_2 and dissolved O in water.

Agriculture and Plant Biochemistry

3544. **Differential loss of dry matter in the laboratory grinding of dried herbage samples.** P. H. Bailey, M. Hughes and A. N. C. McDonald (Nat. Inst. Agric. Engng, Silsoe, Bedford). *J. Brit. Grassl. Soc.*, 1957, **12**, 157–165.—When a hammer mill of the Christy and Norris type is used for milling freeze-dried herbage samples, the loss of protein on a dry-matter basis may be as high as 20%. Investigation showed that freeze-drying produced uncoagulated protoplasmic particles of relatively high protein content, which tended to be lost during milling by passing through or remaining attached to the filter-sleeve. In oven-drying the protoplasm coagulated and adhered to the fibrous particles even during milling, thus reducing the possibility of differential loss of protein. To avoid losses, milled material should be caught in bottles fitted to the mill outlet by a gasket instead of the usual cloth filter-sleeve, and the herbage dust in the working parts should be added to the ground matter. The reduction of air flow when a gasket is used causes the mill to heat up more quickly. NUTR. ABSTR.

3545. **Rapid determination of the major nutrient elements in plants.** R. G. Hutton and P. H. Nye (Univ. Coll., Achimota, Ghana). *J. Sci. Food Agric.*, 1958, **9** (1), 7–14.—The determination of N, P, K, Ca, Na and Mg in a single acid digest of plant material is investigated. *Procedure*—Immediately before digestion dry a sample of ground plant-material (≃ 5 g) overnight in an oven at 105°. Accurately weigh 2·000 g ± 0·005 g of the dried material into a weighed 300-ml digestion flask. Add selenium powder (0·2 g) followed by H_2SO_4 (20 ml). Carry out the normal Kjeldahl digestion. Adjust the weight of the digest to 26 g with H_2SO_4. Add H_2O (20 ml), mix thoroughly and filter through a Whatman No. 50 paper into a 100-ml flask. Rinse the digestion flask with 0·1 *N* H_2SO_4 (5 × 10 ml). Cool the filtrate and adjust the volume to 100 ml. Elements in the digest are then determined by the following procedures. *Nitrogen*—Determine by the Kjeldahl method. *Phosphorus*—Use the method of Kitson and Mellon (*Ind. Eng. Chem., Anal. Ed.*, 1944, **16**, 397), but with half volumes. *Potassium, calcium and sodium*—Determine in an EEL flame photometer against standard soln. *Magnesium*—Transfer an aliquot containing 25 to 150 μg of Mg to a 25-ml flask. Add H_2O (10 ml), starch soln. (1 ml), compensating soln. [$CaCO_3$ 12·5 g, $Al_2(SO_4)_3$ 0·317 g, KH_2PO_4 0·96 g, HCl 2 ml, H_2O to 1 litre] (1 ml), Titan yellow soln. [Titan yellow (0·25 g) in H_2O (500 ml) plus 3 *N* NaOH (3 drops), prepared weekly] (2 ml) and 3 *N* NaOH (3 ml). Mix thoroughly and adjust to volume with H_2O. After 10 min. read the absorption in a 4-cm cell, with a green filter. Determine the content of Mg from a standard curve. An accuracy of ± 5% is claimed. H. B. HEATH

3546. **Micro-determination of silicon in plants.** R. J. Volk and R. L. Weintraub (Chem. Corps Biol. Warfare Lab., Fort Detrick, Frederick, Md., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 1011–1014.—The plant sample is dried, milled and ashed with conc. H_2SO_4. The ingited residue is fused with Na_2CO_3 and the cake extracted with dil. HCl, neutralised and made up to standard vol. An aliquot containing 10 to 50 μg of Si is diluted with water, treated with ammonium molybdate soln., then metol soln. and mixed. The absorption is measured after 3 hr. at 810 mμ. The concn. of Si is derived from calibration curves or a given formula. Variables in the method are discussed; the use of non-siliceous apparatus is obligatory. In normal plant-tissue analysis, phosphate does not interfere. Representative analyses are given.

D. A. PANTONY

3547. **Quantitative determination of ammonia in plants containing tannic substances.** V. R. Popov (A. N. Bakh Inst. of Biochem., Akad. Nauk, SSSR, Moscow). *Biokhimiya*, 1958, **23** (1), 37–40.—The determination of NH_3 in plants containing tannic substances by conventional methods may give incorrect results because of the secondary formation of NH_3 during the reaction, *e.g.*, by air oxidation of polyphenols to quinones, which can cause decomposition of amino acids with liberation of NH_3. The amount of NH_3 liberated depends on the nature and amounts of amino acids and tannic substances present. This may be avoided by removal of the tannic substances, by conducting the distillation of NH_3 in the absence of molecular oxygen, or by adding compounds that are antioxidants for the tannic substances, *e.g.*, ascorbic acid.

C. D. KOPKIN

3548. **The determination of water in the cell material of *Saccharomyces cerevisiae* by the Karl Fischer method.** A. Fiechter and U. Vetsch (Versuchsanstalt für Obst-, Wein- und Gartenbau,

Wädenswil, Switzerland). *Experientia*, 1957, **13** (2), 72–74.—The method described enables the wet and dry weights of small amounts of yeast to be determined. The wet yeast is prepared by filtration under standard conditions, a weighed quantity (50 to 80 mg) is suspended in 10 ml of methanol and titrated with Karl Fischer soln. There is no interference from other substances and the accuracy is ±0·5%. In yeast cells, taken at daily intervals from growing cultures, the dry matter increases from ≃20% to over 30% from the second to the seventh day, respectively. P. NICHOLLS

3549. **Photometric determination of indol-3-ylacetic acid: application to a study of auxin oxidase.** P. E. Pilet. *Rev. Gén. Bot.*, 1957, **64**, 106–122.—A study was made of the development of colour when indol-3-ylacetic acid (**I**) is treated with $FeCl_3$ in the presence of H_2SO_4, including the preparation of reagents, concn., time, temp., and effect of light. The reagent finally developed was 3 ml of 1·5 *M* $FeCl_3$, 60 ml of H_2SO_4 (sp.gr. 1·84) and 100 ml of H_2O, re-distilled and de-ionised. A reference curve is established by using 8 ml of reagent and 2 ml of **I** in a series from 0·25 μg to 10 μg per ml, 40°, and a time of 12 to 20 min. in darkness. Readings are made in a photo-electric colorimeter with a 535-mμ filter. Negligible colour was formed by indol-3-ylcarboxylic, indol-3-ylpropionic and indol-3-ylbutyric acids, indol-3-ylcarboxyaldehyde, DL-tryptophan, indole and skatole. This technique was applied to a study of the **I** oxidase in the meristematic regions of roots of *Lens*. Optimum conditions were pH, 6·2; temp., 36°; temp. of inactivation (time not given), 65°; activators, 2:4-dichlorophenol at 5×10^{-5} *M*, 2:4-dinitrophenol at 8×10^{-6} *M*, 2:4-dinitrocresol at $1{\cdot}1 \times 10^{-6}$ *M*; inhibitors, KCN, CO, $MnCl_2$ at 5×10^{-6} *M*. Enzyme activity was low in young meristems, high in old. CHEM. ABSTR.

3550. **The quantitative determination of 2(3)-benzoxazolinone from rye seedlings.** P. K. Hietala and A. I. Virtanen (Foundation for Chem. Res., Biochem. Inst., Helsinki, Finland). *Acta Chem. Scand.*, 1958, **12** (1), 119–123.—*Procedure*—Homogenised rye seedlings (0·3 to 4 g) are suspended in 15 ml of water and boiled for 20 min. The solution is centrifuged and the residue similarly extracted 4 times more. The combined water extracts are treated with peroxide-free diethyl ether and the ether extract is evaporated to dryness. The residue is dissolved in 1 ml of *cyclo*hexane - ethanol - water (400:80:1) mixture; 0·2 ml of the soln. is placed on top of a 9-mm-diam. cellulose-powder column (preparation described) and eluted with the same mixed solvent, and the fraction passing out when the solvent column has moved between 5·6 and 9 cm is collected. The eluate is made up to a known vol. with ethanol and the extinction measured at 275 mμ. This follows the Beer - Lambert law in the range 50 to 400 μg of 2(3)-benzoxazolinone per 10 ml. The interference of certain other substances may be corrected for by measuring the spectrum in the range 240 to 300 mμ, but, if the absorption due to foreign matter is high, the accuracy is low. E. J. H. BIRCH

3551. **Steam-distillation of ammonia from soil extracts.** D. L. Lyndersen and M. Opem (Sintef. Norges Tek. Høgskole, Trondheim, Norway). *Z. anal. Chem.*, 1958, **159** (5), 339–343.—In order to avoid interference by CO_2 with the titration of ammonia after steam-distillation, $SrCl_2$ is added to the soil extract in the distillation flask. The distillation takes place at pH 8 in borate-buffered soln. Strontium is selected because of the high insolubility of its carbonate. *Procedure*—Shake 10 g of soil with 100 ml of extraction soln. and filter through a dry paper. Place 20 ml of filtrate in a semi-micro distillation flask with 10 ml of buffer soln. (200 ml of 4% boric acid soln. with 50 ml of 0·5 *N* NaOH) and 1 ml of 2 *M* $SrCl_2$. Liberate the NH_3 by steam-distillation into 5 ml of boric acid - indicator soln. (40 g of HBO_3 per litre heated to drive off CO_2; 30 ml of bromocresol green and 10 ml of methyl red soln. are added to the cooled soln.). Titrate with approx. *N*/140 H_2SO_4. D. F. PHILLIPS

3552. **Simple method of determining nitrogen-containing substances in silage. Ammonia nitrogen, water-soluble nitrogen and total nitrogen.** B. Juhász, B. Szegedi and M. Gertner (Tierphysiol. Inst., Agrarwiss. Univ. Budapest, Gödöllö, Hungary). *Z. Tierernähr.*, 1957, **12**, 218–225.—Simple micro-diffusion procedures, suitable for the routine determination of ammonia nitrogen, non-protein nitrogen and total nitrogen in silage are described. The colour produced by Nessler's reagent is measured by a Pulfrich photometer with filter S43, and results can be read from a standard curve. Results agreed well with those of the distillation and ordinary micro-Kjeldahl methods, which are costlier in time and money. Values are tabulated for several kinds of bad silage. NUTR. ABSTR.

3553. **Observations on "crude fibre" estimation by acid digestion.** H. W. Dougall (Grassland Res. Sta., Dept. Agric., Kitale, Kenya). *J. Sci. Food Agric.*, 1958, **9** (1), 1–7.—The solvent action of 0·255 *N*, 1·5 *N*, 3 *N* and 6 *N* H_2SO_4 on grass feeds and faeces, and of 1·5 *N* H_2SO_4 on three other feeds for periods of 0·5 to 3 hr. is studied. The "crude fibre" content is determined as follows. To a 1-litre flask containing 2 g of sample, and provided with a 75-cm air-condenser, add 200 ml of boiling H_2SO_4 of the concn. required. Boil, gently rotating the flask periodically. At the end of the specified time, filter the contents through a 12·5-cm Whatman paper No. 54; wash the residue with boiling water until free from acid, then twice with ethanol and three times with diethyl ether. Dry the filter-paper and residue in an oven at 100° for 18 hr. Transfer the residue to a 7-cm flat-bottomed dish and dry for a further 5 hr., cool and weigh. Ignite for 30 min. at 600°, cool and weigh the ash. Deduct this weight from the weight of the residue. Equations are devised relating the "crude fibre" figure with the concn. of acid and time of digestion. Results are compared with those obtained by acid digestion for 1 hr. with either 5 or 7·5% H_2SO_4 as suggested by Hallsworth [*Agric. Progr.*, 1950, **25** (1), 39]. The pre-extraction of fats with a mixture of ethanol and benzene, and the estimation of the N content of the fibrous residue are proposed. H. B. HEATH

3554. **Spectrophotometric determination of 2-(*p-tert.*-butylphenoxy)-1-methylethyl 2-chloroethyl sulphite (Aramite) residues.** M. E. Brokke, U. Kiigemagi and L. C. Terriere (Oregon Agric. Exp. Sta., Oregon State College, Corvallis, U.S.A.). *J. Agric. Food Chem.*, 1958, **6** (1), 26–27.—This insecticide is extracted from plant samples by benzene or

n-hexane, and the extract is dried with anhydrous Na_2SO_4; on being refluxed with Na *iso*propoxide in acid soln., the Aramite gives ethanediol. This is treated with periodic acid to give formaldehyde, which is determined by the red colour formed on reaction with phenylhydrazine and H_2SO_4 in the presence of periodic acid. Residues as low as 0·1 p.p.m. on 100-g samples were determined. Interference from other pesticides was negligible.
M. D. ANDERSON

3555. **Analysis of mixtures of isomers of demeton.** K. Groves (Dept. of Agric. Chem., State College of Washington, Pullman, U.S.A.). *J. Agric. Food Chem.*, 1958, **6** (1), 30–31.—Mixtures of the thiono and thiol isomers of demeton [diethyl 2-ethylthioethyl phosphorothionate (**I**) and diethyl S-(2-ethylthioethyl) phosphorothiolate (**II**)] are determined by titration with bromate - bromide before and after alkaline hydrolysis. The results of titrations of known mixtures of the isomers and related compounds show the reliability of the method, which was confirmed by determinations of the sulphate formed from the thiono sulphur in **I**. The determination of **I** is affected by the presence of oxidisable impurities; that of **II** is affected only if the reactivity of the impurities with Br is changed by alkaline hydrolysis. M. D. ANDERSON

3556. **Spectrophotofluorimetry for pesticide determinations.** I. Hornstein (Entomology Res. Div., U.S. Dept. of Agric., Beltsville, Md., U.S.A.). *J. Agric. Food Chem.*, 1958, **6** (1), 32–34.—Figures are given for the wavelengths of u.v. light giving maximum activation and fluorescence peaks for the following pesticides, in methanol or water at optimum pH—Guthion [OO-dimethyl S-(4-oxo-3H-1:2:3-benzotriazine-3-methyl) phosphorodithioate], Potasan [O-(4-methylumbelliferone) OO-diethyl phosphorothioate], warfarin, piperonyl butoxide {α-[2-(2-butoxyethoxy)ethoxy]-4:5-methylenedioxy-2-propyltoluene}, *n*-propyl isome {dipropyl 5:6:7:8-tetrahydro-7-methylnaphtho[2:3]-1:3-dioxole-5:6-dicarboxylate}, indol-3-ylacetic acid and naphthalene-acetic acid and -acetamide. Other pesticides examined did not fluoresce. M. D. ANDERSON

See also Abstracts—**3388**, Determination of maleic hydrazide in plant-tissue extracts. **3433**, Determination of ^{90}Sr and ^{140}Ba in vegetation, etc. **3534**, Determination of vitamin B_{12} in cattle faeces. **3540**, Analysis of radioactivity of soils. **3567**, Carrier pptn. of trace elements prior to analysis.

5.—GENERAL TECHNIQUE AND LABORATORY APPARATUS

General

3557. **A "micro" homogeniser for small-volume samples.** H. A. Went (Calif. Univ., Berkeley, U.S.A.). *Biochim. Biophys. Acta*, 1958, **27** (1), 165–167.—The design, construction and performance of a high-speed homogeniser for use with biological samples (2·5 to 7 ml) are given and discussed.
W. H. C. SHAW

3558. **A convenient dropping-funnel for tracer work.** S. Kveder (Tracer Lab., Inst. "Rudjer Bošković," Zagreb, Yugoslavia). *Croat. Chem. Acta*, 1958, **30** (1), 103–104.—The dropping-funnel described has a ground-ball joint instead of the conventional stopcock. The following properties make it particularly suitable for radioactive tracer work—perfect sealing without greasing, the soln. is in contact with glass only, all parts are easily dismantled and may be cleaned separately, and construction is relatively simple. S. M. MARSH

3559. **Vaporimetric molecular-weight determination apparatus. Microchemical apparatus. Group K: Accessories for physical gravimetric methods.** British Standards Institution (2 Park St., London). B.S. 1428: Part K1, 1958, 11 pp.—Components are specified and illustrated for an apparatus (Bratton and Lochte type) for the vaporimetric micro-determination of mol. wt. by the method described by Colson (*Analyst*, 1952, **77**, 139). O. M. WHITTON

3560. **Apparatus for the delivery of measured amounts of gases.** V. Kaláb and J. Pinkava (High School of Chem. Technol., Prague). *Chem. Listy*, 1958, **52** (1), 156–158.—The new apparatus ensures a constant composition and constant flow of the gaseous mixture over long periods even when there are pressure changes in the apparatus. J. ZÝKA

3561. **Electrolytically controlled device for dispensing liquids.** R. J. Heckly (Naval Biol. Lab., Univ. of Calif., Berkeley, U.S.A.). *Science*, 1958, **127**, 233–234.—A compact instrument is described by which the production of gas by electrolysis of water is used for dispensing soln. aseptically at any desired rate by regulation of the electric current. It has been applied to delivering liquids for continuous-flow electrophoresis, for compensation in a Tiselius apparatus, and for controlling flow-rates in ion-exchange or chromatographic separation.
H. F. W. KIRKPATRICK

3562. **Apparatus for the determination of uranium with ether extraction.** Z. Nagy and G. Almássy (Inst. for Med. Chem., Med. Univ., Debrecen, Hungary). *Magyar Kém. Foly.*, 1957, **63** (12), 359–360.—The separation of uranium from large amounts of other metals before colorimetric determination (*cf.* Almássy *et al.*, *Anal. Abstr.*, 1956, **3**, 1344) depends on ether extraction. An apparatus with interchangeable glass joints is described, by means of which the ether layer from the separating funnel is sucked into a flask, from which the ether is then distilled off in a vacuum. This process is repeated three times, and the residue in the distilling flask is then ready for determination.
A. G. PETO

3563. **An improved technique for spot testing, using a Cellophane membrane.** B. Camber (27, Wimpole St., London). *Nature*, 1958, **181**, 124.—For the detection of aldehydes and ketones by the formation of salicyloylhydrazones, a test-tube containing the (volatile) carbonyl compound is covered with a piece of non-waterproof Cellophane previously wetted with 5% aq. acetic acid, which is secured by a rubber band. A drop of a satd. soln. of salicyloylhydrazide is then placed on the Cellophane, which it permeates to meet the carbonyl compound permeating by volatilisation in the opposite direction. For non-volatile substances, the soln. in 5% acetic acid is placed in the tube, the under surface of the Cellophane is wetted with it by inverting the tube, and the reagent is applied as before. The colours are more easily seen and

compared than when developed on filter-paper, and the method is generally applicable to reactions that take place in aq. soln. R. E. ESSERY

3564. **Trace analyses by gas chromatography.** C. E. Bennett, S. D. Nogare, L. W. Safranski and C. D. Lewis (E. I. du Pont de Nemours & Co. Inc., Wilmington, Del., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 898–902.—Trace components in the range 1 to 200 p.p.m. in organic mixtures can be determined by a combination of a thermal conductivity detector, with thermistors, and an amplifier. The techniques necessary for obtaining low noise and drift levels with this combination are described. Specific applications described include the separation and determination of 70 p.p.m. of *iso*propyl alcohol in benzene, the determination of 5 p.p.m. of benzene and 40 p.p.m. of *cyclo*hexanol in toluene, and the determination of 38 p.p.m. of water in methanol. K. A. PROCTOR

3565. **High-temperature gas-chromatography apparatus.** S. D. Nogare and L. W. Safranski (E. I. du Pont de Nemours & Co. Inc., Wilmington, Del., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 894–898.—The relatively simple apparatus described is used for the qual. and quant. resolution and determination of high-boiling organic mixtures. Good resolution was attained in the temp. range 150° to 350° on relatively short columns, with silicone grease or linear polyethylene as the stationary phase. Platinum thermal-conductivity cells are used for detection and are operated at 10° to 100° higher than the temp. of the column to prevent condensation of high-boiling components. Thermal decomposition was minimised by the all-glass construction of the apparatus and by the short time the mixtures were in the columns. K. A. PROCTOR

3566. **A viscometer for rapid measurements of dilute polymer solutions at elevated temperatures.** R. M. Schulken, jun., and M. L. Sparks (Res. Lab., Tennessee Eastman Co., Kingsport, Tenn.). *J. Polym. Sci.*, 1957, **26**, 227–230.—By means of viscometers of the type described and illustrated, an operator can deal with 100 samples in 8 hr., measuring 3 efflux times per sample (precision $= \pm 0{\cdot}05$ sec.) and calculating the results. The 95% confidence limits [*sic*] for a given polymer with an inherent viscosity of $\simeq 1{\cdot}0$ are $< 0{\cdot}02$ decilitre per g. B. J. WALBY

Optical

3567. **Carrier precipitation of trace elements prior to spectrographic analysis.** B. E. Hankins (Univ. Missouri, Rolla, U.S.A.). *Dissert. Abstr.*, 1957, **17**, 2148.—Traces of Cu, Co, Zn, Mo and Ni may be concentrated before spectrographic analysis without loss by the addition of aluminium, oxine, tannic acid and thionalide to the wet-ashed soln. of a 10-g plant sample adjusted to pH 5·2 with ammonium acetate buffer. N. E.

3568. **Spectroscopic studies on rare-earth compounds. I. Instrumentation.** P. Krumholz (Res. Lab. of Orquima S.A., Sao Paulo, Brazil). *Spectrochim. Acta*, 1958, **10** (3), 269–273.—A grating spectrophotometer for the range of 360 to 950 mμ, with a band-width of < 1 A and a wavelength-setting reproducible within $\pm$ 0·1 to 0·3 A, is described. The instrument has a mounting with movable entrance or exit slit, and the wavelength-setting is controlled accurately with a tangent micrometer-screw drive operating on a lever arm. The wavelength calibration changes by < 2 A after 1000 hr. operation. Rapidity of measurement is ensured by a simple device whereby blank readings can be kept constant within 1% over ranges of 500 to 2000 A. W. J. BAKER

3569. **Calibration of infra-red prism spectrometers.** D. A. Dows (Dept. of Chem., Univ. of Southern California, Los Angeles, U.S.A.). *J. Opt. Soc. Amer.*, 1958, **48** (1), 73.—Revised values of frequency are given for a number of HCl spectrum lines proposed by Downie *et al.* (*J. Opt. Soc. Amer.*, 1953, **43**, 941) as frequency standards suitable for the calibration of i.r. prism spectrophotometers. B. S. COOPER

3570. **Simple mulling technique for the preparation of samples for infra-red spectroscopy.** A. Crook and P. J. Taylor (Res. Dept., Tootal Broadhurst Lee Co. Ltd., Manchester). *Chem. & Ind.*, 1958, (4), 95.—Squares (4 in.) of $\frac{1}{4}$-in. plate-glass are ground together with 220-mesh carborundum powder until they are uniformly rough and then rubbed together with a few drops of liquid paraffin until no more glass powder is produced. Powder or cryst. material is mulled in 30 sec. by placing a little between the plates with a few drops of liquid paraffin or hexachlorobutadiene and rubbing manually between the plates. Textile yarns are first cut to 0·5 to 2 mm. Plastic or rubbery materials require the addition of a little NaCl as abrasive and diluent. The plates are cleaned by mulling sodium carbonate and water between them, and washing under the tap. The plates can also be used to re-surface scratched rock-salt plates. Examples of i.r. spectra with fibres and rubber mulled in this way are shown. E. J. H. BIRCH

3571. **Improved optical null infra-red spectrophotometer.** L. W. Herscher, H. D. Ruhl and N. Wright (Dow Chemical Co., Midland, Mich., U.S.A.). *J. Opt. Soc. Amer.*, 1958, **48** (1), 36–42.—A new i.r. spectrophotometer is described that embodies certain special features which give improved performance, particularly in the field of accurate quantitative chemical analysis. These features are —an arrangement for by-passing the sample cell with a small amount of radiation in order to provide a dynamic balance at zero transmission, a means for automatic gain control of the beam comparison signal amplifier during differential runs, a clutch to expand pen-travel to full scale for the transmission range 70% to 100%, improved scan speed control, and a non-linear ($\lambda^{0{\cdot}5}$) wavelength scale. The performance of the instrument is illustrated by recordings of spectra showing various applications. B. S. COOPER

3572. **Anomalous infra-red spectra of solids in the potassium bromide disc technique.** J. B. Jensen (Specialitetskontrollen, Brønshøj, Copenhagen, Denmark). *Dansk Tidsskr. Farm.*, 1958, **32** (1), 26–27.—Reductions in the use of the spectra and in the extinction values are observed for barbituric acids, steroids, and some alkaloids. The anomalies are observed after the mixtures of the test samples with potassium bromide have been pulverised too vigorously. A short heating of the discs at 100°, or keeping for a longer time at room temp., restores the normal characteristics of the spectra. The anomalous spectra are probably caused by the dissolution of the substances in the water contained in the potassium bromide. P. S. ARUP

3573. **Improved infra-red absorption spectra hygrometer.** R. C. Wood (Mechanical Div., General Mills, Inc., Minneapolis, Minnesota, U.S.A.). *Rev. Sci. Instrum.*, 1958, **29** (1), 36–42.—Two narrow bands, one centred near 2·60 μ, the other near 2·45 μ, traverse a 12-in. path through the sample gas. Only the 2·60-μ band is absorbed by water vapour, and the ratio of the intensities of the two transmitted bands is measured by maintaining the ratio at unity by means of a servo-operated glass wedge. G. SKIRROW

3574. **Recording microwave hygrometer.** J. B. Magee and C. M. Crain (Elect. Engng Res. Lab., Univ. of Texas, Austin, U.S.A.). *Rev. Sci. Instrum.*, 1958, **29** (1), 51–54.—By means of a cavity resonator, the contribution of water vapour to the refractive index of air is measured. The device can be extended to the measurement of degree of contamination of one gas by another. G. SKIRROW

3575. **Self-balancing laboratory differential refractometer.** C. J. Penther and G. W. Noller (Shell Development Co., Emeryville, Calif.). *Rev. Sci. Instrum.*, 1958, **29** (1), 43–46.—The instrument described is capable of covering a range of 0·0175 R.I. unit and has a stability and accuracy within 5×10^{-7} R.I. unit. G. SKIRROW

Thermal

3576. **Automatic cryoscopic determination of molecular weights.** E. L. Simons (General Electric Res. Lab., Schenectady, N.Y., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 979–982.—The apparatus described is a completely automatic recording one, in which a thermistor is used as the temp.-sensitive element. Supercooling is automatically controlled. A continuous curve of voltage *vs.* time is obtained and makes extrapolation for freezing-points more accurate. K. A. PROCTOR

Electrical

3577. **Instrumental methods of derivative polarography.** M. T. Kelley and D. J. Fisher (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 929–932.—A dropping mercury electrode polarograph is described for use with dil. soln. (10^{-4} to 10^{-5} *M*). Derivative polarograms are recorded by using a diode filter with a parallel-T filter to remove drop oscillations. The waves show slight dissymmetry, but permit good resolution of half-wave potentials as close as 60 mV. K. A. PROCTOR

3578. **Polarography at very negative potentials. Improvement of polarograms by use of NN-dimethylformamide and tetrabutylammonium iodide.** F. L. Lambert (Occidental College, Los Angeles, Calif., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 1018.—The use of dimethylformamide and tetrabutylammonium iodide for polarography at very negative potentials is recommended because very few erratic drops then occur, no slight imperfections in the envelope of the polarographic curve can be detected, and the capillary stays clean for months if it is stored in pure dimethylformamide. K. A. PROCTOR

3579. **Electrometric titrations with the gold electrode.** H. Khalifa and A. M. Daess (Chem. Dept., Cairo Univ., Egypt). *Z. anal. Chem.*, 1958, **159** (4), 272–277 (in English).—Massive gold has been shown to be a suitable substitute for platinum in electrometric titrations. Results obtained with polished gold electrodes and platinum electrodes for various neutralisation processes and redox systems are compared and show good agreement. Gold electrodes cannot, however, be used for the determination of hydrogen ion concentration. S. M. MARSH

3580. **Chelometric titrations with potentiometric end-point detection. Mercury as pM indicator electrode.** C. N. Reilley and R. W. Schmid (Dept. of Chem., Univ. of N. Carolina, Chapel Hill, U.S.A.). *Anal. Chem.*, 1958, **30** (5), 947–953.—A comprehensive physico-chemical study is made of the mercury electrode used in determination of cation activities by e.m.f. measurements. In particular, a theoretical survey is made of the effect of pH and complexing agents and a practical examination of the effect of pH (and buffering), complexing agents, interferences and individual metals on the potentiometric titration curves. It is claimed that such studies allow prediction of titration conditions, and the behaviour of new complexing agents. D. A. PANTONY

3581. **Preparation of carrier-free vanadium, scandium and arsenic activities from cyclotron targets by ion exchange.** U. Schindewolf and J. W. Irvine, jun. (Lab. for Nucl. Sci., M.I.T., Cambridge, Mass., U.S.A.). *Anal. Chem.*, 1958, **30** (5), 906–908.—Titanium and germanium targets are dissolved in HF - H_2O_2 or HF - HNO_3 mixtures and SO_2 is used to give V^{IV} and As^{III}. In dil. (0·5 to 2·5 *M*) HF soln. the complex fluorides of the metals are added to an anion-exchange column in its fluoride form. Vanadium and As pass through and are thus separated from Sc and Ti and from Ge and Ga, respectively, which are retained on the resin. When the column is eluted with 15 *M* HF, the Sc is eluted before the Ti. D. A. PANTONY

3582. **An ionisation chamber for the study of plutonium hexafluoride.** J. W. Codding, jun. (Knolls Atomic Power Lab., Schenectady, N.Y.). *U. S. Atomic Energy Comm.*, Rep. KAPL-1759 1957, 23 pp.—An ionisation chamber constructed of nickel and ceramic alumina is shown to be suitable for the determination of concn. of PuF_6 gas. Calibration was carried out for PuF_6, He and F in the pressure range 0·1 to 100 mm of mercury. The total saturation ion current is not a linear combination of the ion currents caused by each gas separately, and hence a detailed calibration is required for the precise analysis of gas mixtures. Because of the progressive deposition of the decomposition products of the PuF_6 on the walls, the system must be re-calibrated regularly with a gas such as He. Equations developed for the relation between ion current and pressure give results in reasonable agreement with experiment. CHEM. ABSTR.

3583. **Separation and applications of stable isotopes. II. Preparation of gas samples for mass-spectrographic determination of isotope abundances.** I. Maass (Inst. für angew. phys. Stofftrennung, Leipzig, E. Germany). *Chem. Tech., Berlin*, 1958, **10** (1), 17–22.—Methods for the mass-spectrographic analysis of the relative abundances of various isotopes are reviewed. Some typical isotope analyses of hydrogen, carbon, nitrogen and oxygen are described, with accounts of the reactions involved. Appropriate types of apparatus for each analysis are reported. (19 references.) C. A. FINCH

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	A
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
concentrated	conc.
concentration	concn.
constant	const.
corrected	(corr.)
crystalline / crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_λ^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_F
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{2+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

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ANALYTICAL ABSTRACTS

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